Quality Assurance Project Plan

For

Yakima Basin Nitrate Study Phase 3 – Comprehensive Analytical Source Tracer Sampling April 2010 Sampling Event

Yakima County, Washington

U.S. EPA Region 10
1200 6th Ave
Seattle, WA 98101

April 8, 2010

QAPP Approvals:

_____ Date: _____
EPA Field Sampling Manager, Curt Black

_____ Date: _____
RARE Project Applicant, Theogene Mbabaliye, Ph.D.

_____ Date: _____
EPA Health, Safety and Environmental Management, Cathe Bell

_____ Date: _____
EPA Regional Quality Assurance Manager, Gina Grepo-Grove

1.0 Project Management Elements

1.1 Table 1 Distribution List

Copies of the completed/signed project plan should be distributed to:

Name	Affiliation	Phone/E-mail
Curt Black	EPA Sampling Project Manager	206 553-1262
Gina Grepo-Grove	EPA Regional Quality Assurance Manager (RQAM)	206 553-6395
Theogene Mbabaliye	RARE Project Applicant	206 553-6322
Mike Cox	OEA REU Manager	206 553-1597
Eric Winiecki	EPA Office of Water Contact	206-553-6904
Gerald Dodo	Manchester Lab Contact	360-871-8728
Stephanie Harris	Lead Microbiology	360-871-8710
Cathe Bell	EPA Health and Safety	206 553-0308
Sandra Halstead	EPA Prosser Place Based	509-786-9225
Steve Hutchins	Ada Lab, RARE Project Manager	580-436-8563

1.2 Project/Task Organization

This section identifies the personnel involved in the Yakima County Phase 3 - Comprehensive Analytical Source Tracer Sampling Project and defines their respective responsibilities.

Curt Black, EPA Analytical Tracer Project Sampling Manager - Will be the responsible official for this sampling project. Responsible for overseeing the overall sampling and scheduling the work required to complete this project among the Offices and partner Agencies involved. Curt can be reached at 206-553-1262 or in the field at 206 755-4541.

Gina Grepo-Grove, Regional Quality Assurance Manager (RQAM) - The EPA RQAM or her staff will be responsible for reviewing and approving the QA Plan. She may also provide technical input on proposed sampling design, analytical methodologies, and data review or may assist with coordinating laboratory services. Gina is independent of the unit generating or using the data. Gina can be reached at 206-553- 1632. Don Matheny of the QA Unit is the specific contact who coordinated with Manchester and can be reached at 206-553-2599

Brent Richmond, Field Support Coordinator - Will be responsible for preparing needed sampling equipment and sampling containers supplied by MEL. Brent will report to the Project Manager. Brent can be reached at 360-871-8711. Note, some sample containers are being provided by the EPA, Ada Oklahoma lab because of their access to an annealing oven for baking the containers used in the trace-organic analysis.

Gerald Dodo, Manchester Laboratory Contact - Answers questions on lab capability and capacity. Gerald can be reached at 360-871-8728.

Bethany Plewe, Regional Sample Control Center, will be providing sample numbers on a weekly basis and scheduling samples into the Manchester Lab. Bethany can be reached at 206-553-1603.

Steve Hutchins, Regionally Applied Research Effort (RARE) Project Manager, will be arranging laboratory capacity for the trace organic analysis, hormones, isotopes. He is also administering the RARE project through ORD. Steve can be reached at 580-436-8563.

Karen Norton, ESAT Manchester Laboratory Contractor, will be receiving the Manchester lab samples. Karen can be reached at 360-871-8760.

1.3 Problem Definition/Background

1.3.1 Background

Ground-water sampling data from 1990-2008 in the Lower Yakima Valley of Washington State has shown as many as 12% of area wells to be contaminated above the drinking water standard for nitrate (10 mg/L) and about 20% to demonstrate bacterial contamination (Options Paper, 2009) These wells tend to be shallow and in many cases are primarily used by tribal members of the Yakima Nation, or Spanish speaking families participating in the local agricultural economy. However, these wells may be used by anyone in the community not served by a public water-supply system.

Sampling efforts conducted to date have focused on nitrate primarily because EPA has a national standard for this compound. However, utilizing resources made available in the Regionally Applied Research Effort (RARE) program, we propose a sampling approach which, over the next few months, will extend far beyond nitrate. In this way we hope to identify the causes of high nutrient levels in the ground water of the Lower Yakima Valley. In this program we hope to link the land-uses which are the source of nitrate with its fate in local drinking water wells.

EPA is going into the field to answer questions raised by the community in the Yakima Valley. We are using funding from the RARE Program, as well as other Region 10 funding sources. At a meeting in December, 2008, EPA was advised that community members want to know what is in their drinking water. If it is contaminated, they want to know the source of that contamination. While no systematic testing of private drinking water wells is done by any level of government, sampling for nitrate has been conducted in the lower valley for decades. Despite this testing, today Yakima residents still have no way to link land uses to observed adverse effects in the quality of their ground water.

With this study, EPA proposes an even-handed assessment of potential sources of nitrate by working to analytically detect trace concentrations of compounds released with the nitrate. We will also look at other chemical changes which have been used to link up-gradient land use to effects in drinking water wells. EPA hopes to assess which

activities on the landscape are contributing excess nitrate to shallow ground water. In this way we can determine which practices or activities are protective of continuing ground-water quality, and which activities are threats to the ground-water resource. In addition, the sampling from this project will provide enforcement-quality data for nitrates and bacteria that could be used in later enforcement actions.

Potential sources of nitrate include rural residential septic systems, low-density animal husbandry, application of chemical fertilizers to agricultural crops, application of manure to agricultural crops, spray-field application of nutrient-rich waste waters, and confined animal operations, either the operations themselves, manure storage facilities, or the lagoons which contain liquid effluent from those operations.

In order to develop a linkage between the high nitrate observed in a private drinking water well and the land use which is adversely affecting ground-water quality it will be necessary to look beyond nitrate. Analytical methods are available to detect a wide range of organic compounds which originate with the nitrate and which may provide a linkage back to the source. By looking at the agricultural chemicals, the hormones or the personal care products traveling with nitrate, we hope to link and identify the farming practice, the confined animal feeding operation or the rural residential septic system which is the source of the nitrate, respectively. Many of these trace organics can be detected at exquisitely low concentrations – some as low as single digit parts per trillion.

These hormones, pesticides, personal care products and pharmaceuticals are widely used by either humans, used for veterinary care of livestock or applied to crops. Additionally, isotopic techniques are available to characterize the nitrogen and oxygen components of the nitrate molecule which may be useful to trace the nutrient back to its source. Finally, for wells with significant fecal coliform contamination, we can employ microbial source tracking to determine if the source of the coliform was human or ruminant.

1.3.2 Objectives/Scope

This project is organized into phases. Phase 1 included the development of a GIS tool to organize the large amount of historical information and allow the examination of the landscape for spatial patterns in that data. This tool, which continues to improve, is in use and serving the project well.

The objective of Phase 2 was to evaluate whether there are drinking water wells with nitrate levels over the MCL down gradient of potential nitrate contaminant sources. This enabled us to select nitrate sources and sampling sites for further source characterization in Phase 3. Through our Safe Drinking Water Act authorities, the MCL exceedances documented in Phase 2 will become the basis of our access to source areas for characterization in Phase 3. The Phase 2 data will further help management evaluate whether there is a basis for enforcement action. The sampling from Phase 2 supplied enforcement-quality data for use in later enforcement actions.

The objective of Phase 3 is to conduct research/sampling under a RARE grant to test techniques which may improve our abilities to link specific human practices to high nitrate levels in groundwater and private wells. Phase 3 will select the highest nitrate

concentration wells identified in Phase 2 for re-sampling. Phase 3, the Regionally Applied Research Effort (RARE) sampling, will analyze for a wide range of potential tracing or linking compounds which may be traveling with the nitrate. These compounds would then potentially identify, or link back to, the land use from which the nitrate came. These compounds include estrogens, androgens, veterinary and human antibiotics, agricultural chemicals, personal care products and human medications and compounds such as caffeine or ibuprofin. Additionally, analyses will be performed for common degradation byproducts from the compounds above. Isotopic analysis of the nitrogen in nitrate and ammonia and the oxygen in nitrate as well as determination of "time-since infiltration" will be performed using sulfur hexafluoride.

The use of private domestic water supply wells requires contacting individual home owners and requesting access to sample their drinking water. It further involves using wells installed for another purpose for use as groundwater monitoring wells. These wells may not be screened at horizons where they optimally intersect the water we seek to monitor. However, the alternative of either direct-push geoprobe style investigatory soil borings or dedicated well installations is not feasible given our interest in timely collection of this data and the resources available.

Phase 3 is attempting to demonstrate the potential to use low concentrations of trace organic compounds to link land use to observed nitrate contamination. These organic compounds are subject to microbial degradation. Further, they tend to sorb to the organic carbon in the porous media of the aquifer matrix. This can significantly retard the movement of the organic molecules with respect to the movement of groundwater or nitrate. The specification of very low detection limits for many of the organic analyses in this project are hoped to overcome these limitations. Phase 3 will also provide enforcement-quality data.

Phase 3 will include revisiting the domestic wells which tested highest for nitrate in Phase 2. Phase 3 also involves moving up-gradient from those high nitrate wells to sample water from a variety of sources such as dairies or agricultural crop land. Samples will be obtained from three small sewer treatment plants to allow characterization of the typical compounds introduced with rural septage.

A Phase 4 is possible using dedicated transects of geoprobe borings and other appropriate technology for accurate characterization of ground water impacts from the areas under investigation. The objective of Phase 4 is to evaluate data from Phase 3 to determine next steps to better control nitrate contamination including potential enforcement actions.

2.0 Project/Task Description and Schedule

2.1 Project/Task Description

2.1.1 Phase 1 – The project team has developed a Geographic Information System (GIS) application that was used to identify sample sites for Phase 2 of the project. The locations selected were locations with the greatest potential for impacts to the aquifer as well as the greatest concentration of domestic supply wells for monitoring aquifer

impacts. The tool incorporates the majority of known nitrate, Coliform, ammonia, and general chemistry data from the lower Yakima Valley. It also includes the locations of wells, well analytical results, home tracts with septic systems, land elevation, elevation of ground water surface, aquifer vulnerability to contamination, crop type, estimated fertilizer application rates, dairy and other animal feeding operation (AFO) locations, roads and an aerial photo layer. As the Phase 2 data became available, it was included.

The project team utilized the following criteria when sites were selected for sampling in Phase 2

Rural Residential Septic Systems:
Select an area with a high concentration of homes not served by sanitary sewer. Select a community with a location on the aquifer such that ground-water flow can be expected to be consistent from season to season and is predictable in direction. Select sites which present as little as possible up-gradient contributing sources of nitrate. Select sites with a linear array of homes using individual domestic water supply wells along the down-gradient side of the community. Select sites with demography similar to the sewered control community from which specific trace-organic analytes which will be used to characterize rural residential septage will be selected. Select sites with a history of down-gradient detections of nitrates in excess of the MCL.

Agricultural Crop Land Sites:
Select an area with a history of use for crops requiring a high fertilizer application rate. To the extent possible, select a crop with a history of heavy accessory agricultural chemical use. Select a tract with a location on the aquifer such that ground-water flow can be expected to be consistent from season to season and is predictable in direction. Select sites which present as little as possible another different up-gradient contributing sources of nitrate. Select sites with a linear array of homes using individual domestic water supply wells along the down-gradient side of the crop land. Select sites with a history of down-gradient detections of nitrates in excess of the MCL.

Animal Feeding Operations (AFOs)
Select an area with a high concentration of animal units per unit area of available land. Select an AFO with a location on the aquifer such that ground-water flow can be expected to be consistent from season to season and is predictable in direction. Select sites which present as little as possible another up-gradient contributing sources of nitrate. Select sites with a linear array of homes using individual domestic water supply wells along the down-gradient side, or sides, of the area of interest. Select sites with a history of compliance issues or community complaints, other factors being equal. Select sites with a history of down-gradient detections of nitrates in excess of the MCL.

2.1.2 Phase 2 –A series of public meetings, newspaper articles and radio announcements notified the community of the Phase 2 work. Two-person teams consisting of two EPA employees trained for the project were used. At all times one of the team members was a credentialed officer of the Agency. The other team member also met EPA requirements for such field work.

Sample teams established contact with the homeowner, collected a GPS location from the well, and filled in a data form developed by the EPA. The teams used a nitrate test

strip to evaluate whether the water exceeded the maximum contaminant limit (MCL) for nitrate.

If the test strip indicated the water may exceed the MCL, the sampler collected samples for analysis for nitrate-N by EPA's Manchester Environmental Lab (MEL). This same individual collected a sample for enumeration/quantification of total coliform; with fecal coliform or E. coli analysis conducted if the sample was total coliform positive.. For wells with coliform bacteria exceeding a threshold value (>14cfu/100ml), the mobile lab was used for follow-up microbial source tracking analysis. For low oxygen or oxidation/reduction potential wells, a TKN sample was taken to account for nitrogen in forms other than nitrate.

During the 2 weeks EPA was in the field, 337 homes were visited. Of those, just over 20% were found to exceed the MCL for nitrate. Eight wells were found which had fecal coliform bacterial contamination or contamination with E.coli. Of the exceedances for nitrate, values as high as 53 mg/l NO3-N were observed, or just over five times the MCL of 10 mg/l NO3-N.

2.1.3 Phase 3 – The subset of homes screened in Phase 2 which were among the highest in nitrate have been selected, as part of the RARE project, to be analyzed for an extensive list of analytes along with general water chemistry parameters. The breakdown of analytes by source is included below in Tables 7 through 18. Domestic water-supply well sampling will be augmented with sampling from selected source areas such as dairies, waste-water treatment plants and crop-land sources. The classes of organic compounds in the RARE study include: hormones, antibiotics, trace organics such as personal care product, pharmaceuticals, and pesticides. Additionally we will determine the isotopic makeup of the nitrogen and oxygen in nitrate in a subset of wells for source tracking. We will determine the nitrogen isotopic makeup of ammonia from lagoons and manure. General chemistry data such as major cation and anion analyses should also provide a great deal of information about the drinking water allowing typing, tracking and linking to specific sources. As many as 30 samples will be collected for time-since-infiltration determinations – or "age dating" of the water. In this way we can determine how far back we are looking in time as we measure chemical conditions in the aquifer. The use of Sulfur Hexafluoride (SF6) as an age dating tool is well matched to the aquifer conditions we observed in Phase 2. SF6 can persist unaltered by low oxidation-reduction potentials which were observed over a large portion of the lower valley. Techniques such as chloro-flurocarbon typing are not typically successful in areas with low oxygen. The time period expected for SF6 is 0 to 40 years before present to within 0.5 to 1 year .

Table 2: Phase 3 AFO Samples

Sampling Location	# samples per facility	Analytes
Upgradient wells	1	MEL General water chemistry, MEL Pesticides, MEL Bacteria, ADA Hormones, UNL Isotopes, USGS Age Dating, USGS Trace Organics, UNL Hormones, UNL Antibiotics, UNL Trace Organics, UNL Extra
Facility supply well (well providing the majority of water used by facility)	1	MEL General water chemistry, MEL Pesticides, MEL Bacteria, ADA Hormones, UNL Isotopes, USGS Age Dating, USGS Trace Organics, UNL Hormones, UNL Antibiotics, UNL Trace Organics, UNL Extra
Manure from storage piles - prior to land application - multi-increment sample of at least 30 subsamples seeking to represent the stock pile - composited and field mixed in stainless bowl)	1	Extraction followed by Analysis: MEL Solid Pesticides, UNL Hormones, UNL Antibiotics, UNL Trace Organics, UNL Extra
Surficial Soil from waste application fields (large area multi-increment sample of at least 30 subsamples seeking to represent the field, composited and field mixed in stainless bowl)	1	Extraction followed by Analysis: MEL solid Pesticides, UNL Hormones, UNL Antibiotics, UNL Trace Organics, UNL Extra
Lagoons – liquids from the freshest source in the lagoon system	1	MEL General water chemistry, MEL Pesticides, MEL Bacteria, ADA Hormones, UNL Isotopes, USGS Trace Organics, UNL Hormones, UNL Antibiotics, UNL Trace Organics, UNL Extra
Lagoons - liquids from the last lagoon prior to pumping onto agricultural land/spray fields	2 samples	MEL General water chemistry, MEL Pesticides, MEL Bacteria, ADA Hormones, UNL Isotopes, USGS Trace Organics, UNL Hormones, UNL Antibiotics, UNL Trace Organics, UNL Extra
Down-gradient wells (Monitoring or Domestic Supply) * With Gas Study where indicated on Sample Count Sheet	3	MEL General water chemistry, MEL Liquid Pesticides, MEL Bacteria, ADA Hormones, USGS Age Dating*, USGS Trace Organics, UNL Hormones, UNL Antibiotics, UNL Trace Organics

Table 3: Phase 3 Crop Land Samples

Sampling location	# Samples	Analytes
Mint Fields Surficial Soil from fields (large area multi-increment sample of at least 30 subsamples seeking to represent the field, composited and field mixed in stainless bowl)	2 fields selected based on Phase 2 domestic wells down-gradient	Extraction followed by Analysis: MEL Pesticides, UNL Hormones, UNL Antibiotics, UNL Trace Organics
Corn Fields Surficial Soil from fields (large area multi-increment sample of at least 30 subsamples seeking to represent the field, composited and field mixed in stainless bowl)	2 fields selected based on Phase 2 domestic wells down-gradient	Extraction followed by Analysis: MEL Pesticides, UNL Hormones, UNL Antibiotics, UNL Trace Organics
Hop Yards Surficial Soil from yards (large area multi-increment sample of at least 30 subsamples seeking to represent the yard composited and field mixed in stainless bowl)	2 yards selected based on Phase 2 domestic wells down-gradient	Extraction followed by Analysis: MEL Pesticides, UNL Hormones, UNL Antibiotics, UNL Trace Organics
Domestic wells selected based on Phase 2 nitrate levels. Duplicate Samples and Gas Study where indicated on Sample Count Sheet	4 households - 1 per house	MEL General water chemistry, MEL Pesticides, MEL Bacteria, ADA Hormones, UNL Isotopes, USGS Age Dating, USGS Trace Organics, UNL Hormones, UNL Antibiotics, UNL Trace Organics

Due to crop rotation, all pesticides/herbicides will be run on all Crop Soil Extracts

Table 4: Phase 3 Rural Septic Samples

Sampling Location	# Sites	Compounds
Small Sewer Treatment Plants	3 (1 per plant)	MEL General water chemistry, MEL Pesticides, ADA Hormones, UNL Isotopes, USGS Trace Organics, UNL Hormones, UNL Antibiotics, UNL Trace Organics
Domestic wells selected based on Phase 2 nitrate levels * With Duplicate Samples and Gas Study where indicated on Sample Count Sheet	4 households - 1 per house	MEL General water chemistry, MEL Pesticides, MEL Bacteria, ADA Hormones, UNL Isotopes, USGS Age Dating, USGS Trace Organics, UNL Hormones, UNL Antibiotics, UNL Trace Organics

Table 5: Schedule of Tasks

Task	Start Date	Completion Date
Phase 3 – QAPP Ver. 1 Review	February 1, 2010	March 31, 2010
Phase 2 – Nitrate Sampling	February 22, 2010	March 7, 2010
Phase 3 – QAPP Ver. 2 Review	March 31, 2010	April 6, 2010
Phase 3 – RARE Sampling	April 12, 2010	April 22, 2010
Laboratory Analysis/QA	April 13, 2010	September 30, 2010
Report Preparation	October 4, 2010	November 8, 2010

3.0 Data Quality Objectives and Criteria for Measurement Data

Data Quality Objectives (DQOs) are the quantitative and qualitative terms field personnel and project managers use to describe how good the data needs to be in order to meet the project's objectives. DQOs for measurement data (referred to here as data quality indicators) are precision, accuracy, representativeness, completeness, comparability, and measurement range. The overall QA objective for analytical data is to ensure that data of known and acceptable quality are provided. To achieve this goal, data must be reviewed for 1) representativeness, 2) comparability, 3) precision, 4) accuracy (or bias), and 5) completeness. Precision, accuracy, completeness, sample representativeness and data comparability are necessary attributes to ensure that analytical data are reliable, scientifically sound, and legally defensible. Each analytical result or set of results generated should be fully defensible in any legal action, whether administrative, civil, or criminal.

Precision: Samples in duplicate will be analyzed on a 10 % frequency (1 per 10 samples collected) for most of the analyses conducted as part of this study. Some analyses are more frequently duplicated with the Gas Study and SF6 analyses being collected and analyzed in duplicate for every sample (100% frequency). The precision is evaluated using the Relative Percent Difference (RPD) values between the duplicate sample results.

Accuracy: For parameters analyzed in the fixed laboratory, accuracy will be evaluated by the use percent recovery (%R) of the target analyte in spiked samples and also the recoveries of the surrogates in all samples and QC samples.

$$\% \text{ Recovery} = \frac{SQ - NQ}{S} \times 100$$

SQ = quantity of spike or surrogate found in sample
NQ = quantity found in native (un-spiked) sample
S = quantity of spike or surrogate added to native sample

Representativeness is the degree to which data from the project accurately represent a particular characteristic of the environmental matrix which is being tested. Representativeness of samples is ensured by adherence to standard field sampling

protocols and standard laboratory protocols. The design of the sampling scheme and number of samples should provide representativeness of each matrix being sampled.

Comparability is the measurement of the confidence in comparing the results of one sampling event with the results of another achieved by using the same matrix, sample location, sampling techniques and analytical methodologies.

Critical measurements for this study include quantification of a wide range of trace-organic compounds concentrations in ground water. Included are androgens, estrogens, veterinary and human pharmaceuticals, personal care products. We will also characterize general ground-water geochemistry at each location sampled and quantify nitrogen and oxygen isotopic makeup of the nitrates in each sample and field parameters.

Phase 3 sampling is intended to provide a linkage between elevated nitrate observed in domestic supply wells and the up-gradient land uses which are responsible for the nitrate. These data will then be used in determining flow within the aquifer and source characterization. The measurement data criteria are typical of enforcement cases with the exception of some of the research-level quantification methods which are not typically available from laboratories. Phase 3 is relying on research labs to provide state of the art analytical methods for hormones and other trace organic compounds at single-digit part per trillion detection limits to part per billion limits depending on the compound. Refer to Tables 7-18 for Method Detection Limits (MDL) and Method Reporting Limits (MRL) for the analytes to be analyzed in this study.

This phase of the research study will be adequately illustrative of aquifer characteristics if data at approximately the MDL Limit listed in Tables 7-18 are provided by each lab. All samples may have an enforcement use and will be collected by an EPA Sampler trained in such sample collection.

Completeness: Completeness is the percentage of valid results obtained compared to the total number of samples taken for a parameter. Since sampling from inspections are usually grab and limited in number of samples, the number of valid results obtained from the analyses are expected to be equal or better than 85%. %Completeness may be calculated using the following formula:

$$\% \text{ Completeness} = \frac{\text{\# of valid results}}{\text{\# of samples taken}} \times 100$$

The QA objectives outlined, above, will be evaluated in conjunction with the data validation process.

3.1 Special Training Requirements/Certification

Field personnel are required to complete the 40-hour Basic Health and Safety training. The personnel will obtain a basic health and safety training certification from the 40-hour training which should be maintained current by attending an 8-hour safety training refresher course every year. The lead sampler will have inspector training and/or

inspector credentials. Furthermore, sampling and sample documentation skills are also assured by the "mentoring" provided by the senior inspectors in the field. The senior worker on each field team will be responsible for all aspects of the collection of all samples.

Additionally, each person participating on a sampling team will undergo a 1 day workshop to go over the objectives, approach, equipment, calibration, data collection, record-keeping, site safety and shipping requirements for this project.

The laboratories performing the sample analysis of drinking water analytes for this program are SDWA certified and/or accredited. Scientists (Microbiologists/Chemists) performing the analytical work for this project have extensive knowledge, skill and demonstrated experience in the execution of the analytical methods being requested.

3.2 Documentation and Records

Complete documentation for field sampling teams may include but is not limited to the following forms:

- Site Sampling data form – one per household, Treatment Plant, AFO or Facility sampled
- Chain of Custody Logs
- Sample Container Labels
- Custody Seals for Shipping Containers
- Photographs, Sketches, Photo Logs or other documentation.

Field Teams will maintain field notes and all documents and data collected will be submitted to the field sampling program manager.

The following documents will be archived at the Manchester Environmental Laboratory or the designated laboratory performing the analysis: (1) signed hard copies of sampling and chain-of-custody records (2) electronic and hard copy of analytical data including extraction and sample preparation bench sheets, raw data and reduced analytical data.

The laboratories will store all sample receipt, sample login, extraction/preparation, and laboratory instrument print-outs and other analytical documentation as per their established SOP.

3.3 Measurement/Data Acquisition

3.3.1 Sampling Process Design

The Phase 3 sampling is attempting to use linking or tracer compounds to evaluate linkage between up-gradient land uses to nitrate in down-gradient drinking water wells. To screen for those compounds, EPA will sample in wells identified as having the highest nitrate concentrations observed in Phase 2 (More than 20% exceeded the drinking-water standard of 10 mg/L nitrate-N). Several wells were seen which

exceeded 50 mg/L nitrate-N. Domestic water-supply well sampling will be augmented with sampling from selected source areas such as dairies, waste-water treatment plants and crop-land sources. The classes of organic compounds in the RARE study include: hormones, antibiotics, trace organics such as personal care product and pharmaceuticals, and pesticides. Additionally we will determine the isotopic makeup of the nitrate in the re-sampled wells for source tracking. The general chemistry data will also be used to type, track and link to specific sources using Piper plots to visualize chemical evolution of ground water as it interacts with various sources. Sulfur Hexafluoride analysis will be used to determine the time-since-infiltration for the ground water. This "age dating" will help evaluate the source of the nitrate contamination. It will also permit evaluation of the potential time lag in improvement in ground-water conditions if land use practices were to change based on the findings of this study.

We are sampling all the wells in this study for the same extensive list of analytes. We have in place both negative and positive controls for the analyses being conducted. We will collect a field-blank while in the lower valley to demonstrate our technique and container set is free of the compounds for which we are analyzing. The laboratories in this study each will be running analytical duplicates and matrix spikes to demonstrate their ability to detect these same compounds in these groundwaters.

Following Phase 2, selected high nitrate wells were evaluated for logical patterns of sources for observed nitrate. In addition to re-sampling the high nitrate domestic water supply wells for a wide range of organic and inorganic compounds, the potential up-gradient sources will be characterized.

Source characterization will differ for AFOs and crop land although the analyses conducted will be the same for all.. For dairies and other AFOs, several samples will be collected from each facility. The lagoon systems will be sampled, on-site wells and manure piles, an up-gradient well and the best available down gradient wells (those showing the highest Phase 2 nitrate values) will all be sampled.

For crop land, three crops were selected based on fertilizer application rates and pesticide usage. Corn fields, hop yards and mint fields were selected based on the amount of fertilizer used in their culture. The samples will be multi-increment samples to provide better homogenization of what is expected to be a very heterogeneous source material.

For rural residential septic characterization, samples will be taken from the influent stream to 3 different small sewer treatment plants in the lower valley. These plants will be selected so as to have a similar demographic in the contributing area as the adjacent non-sewer area. In this way it is not necessary to sample from a specific drain field and a wider range of potential linking organic compounds will be encountered. Four home locations down-gradient of significant concentration of septic systems and which provided high nitrate samples in Phase 2 will be re-sampled in Phase 3 for the same wide range of trace organic compounds, age dating gases and general chemical analytes to provide the strongest basis for comparison with the AFO and crop-land associated wells.

3.4 Sample Collection Procedures

3.5 Health and Safety

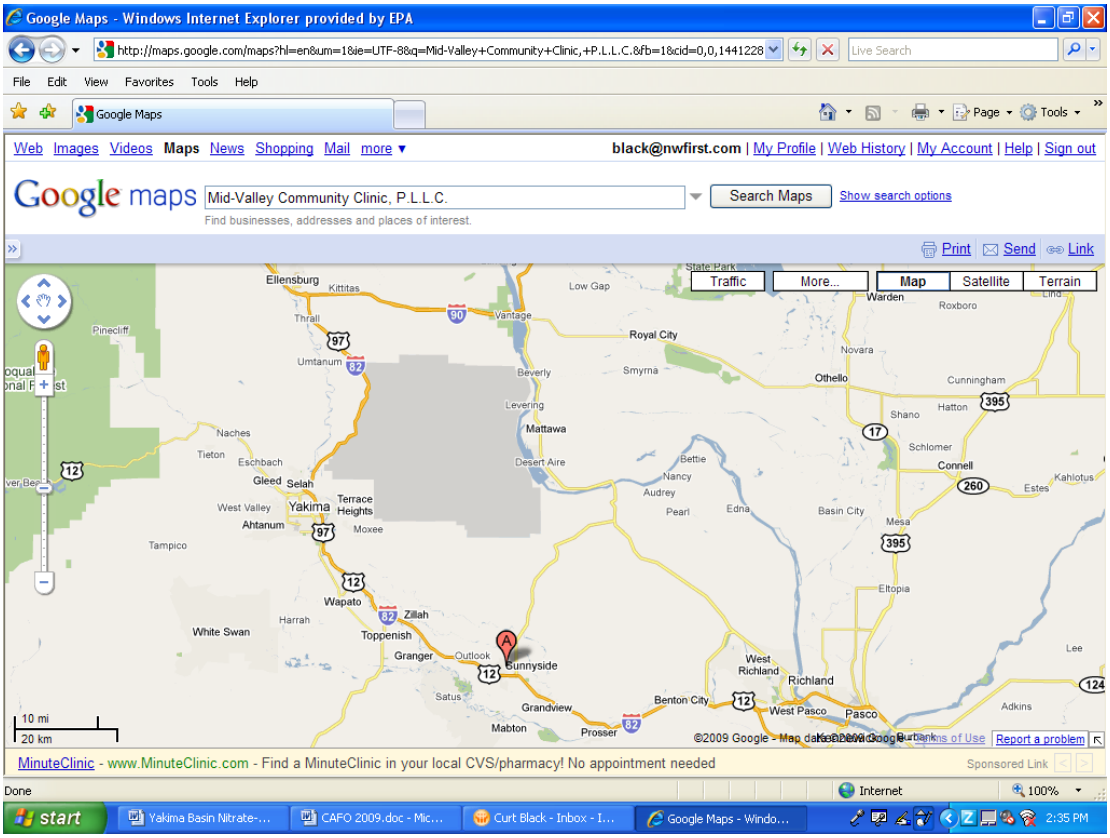
All personnel participating in this project will follow the safety requirements outlined below. Samples collected during this sampling range from drinking water well samples from household taps to composite soil samples from area cropland to influent sewage samples from sewer-treatment plants to lagoon and manure samples on AFOs. All personnel should be aware of the potential hazards associated with the collection, handling, analysis, and disposal of the samples. It is the responsibility of the participating personnel to follow all necessary safety measures and to bring to the attention of the project manager any issues concerning safety.

Job Hazard Assessments have been completed for the mobilization and sampling activities of this project. They are in Appendix 3. As some sampling is occurring in private homes, significant concerns surround approaching a home. These may be in unfamiliar neighborhoods, and may be in the territory of a domestic dog with an attitude. It is similarly possible to encounter home owners who are unsympathetic to our work. If the team encounters a home owner with a strongly negative response, simply withdraw. For work on AFOs we need to remain aware that we may be as dangerous to the livestock as the lagoon sampling might be to us. All sampling materials and PPE entering and leaving the AFO site need be either new or sterilized before and after use with a bleach or equivalent anti-microbial wash down.

Field personnel will have a cell phone for emergency contacts. Cell phone coverage is good in the Lower Yakima Basin. Emergency services can be reached by dialing 911.

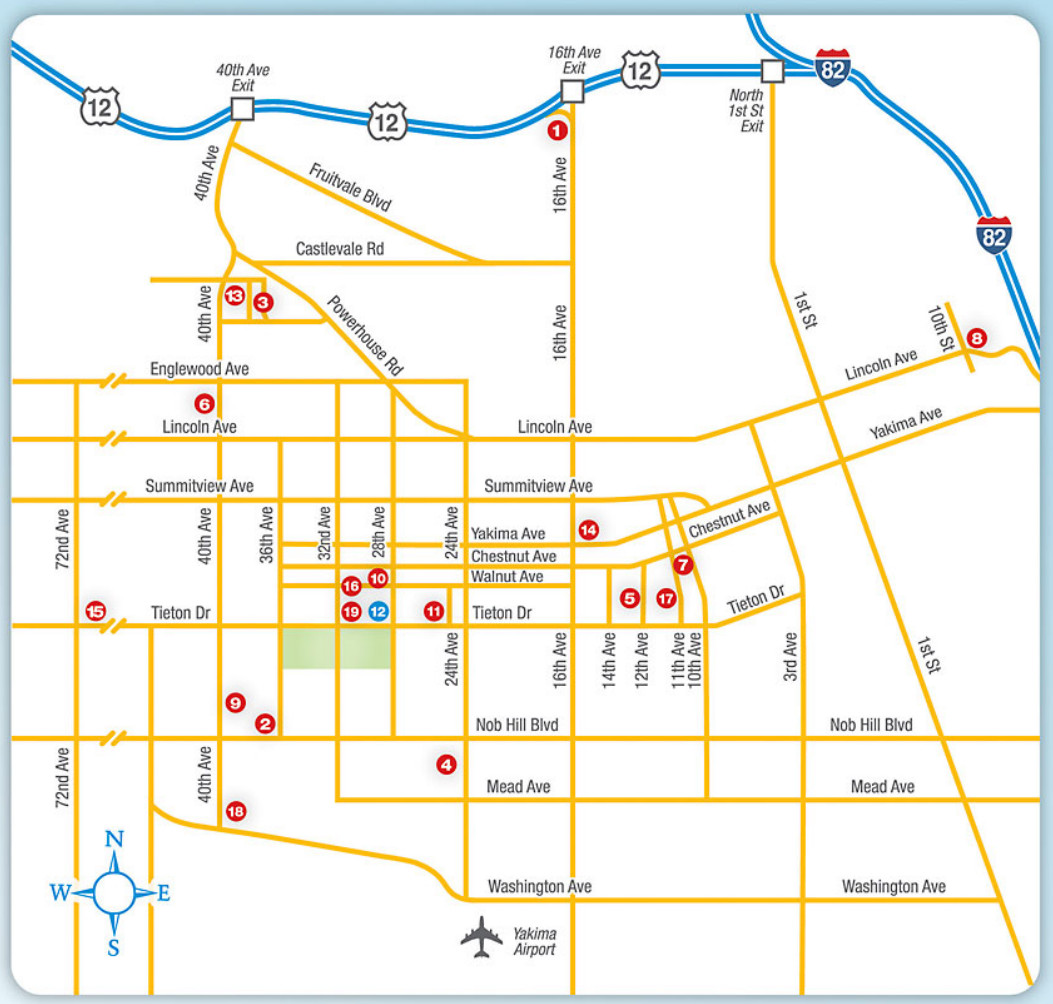
Nearby hospitals include:
Yakima, Washington. Yakima Valley Memorial Hospital • 2811 Tieton Drive • Yakima, WA 98902 • (509) 575-8000
Sunnyside Washington. Mid-Valley Community Clinic •700 South 11th •Sunnyside,WA 98944 • (509) 839-6822

3.6 Valley Overview Map:



See Attached Map Yakima Valley Memorial Hospital, 2811 Tieton Drive, Yakima 509 575-8000

Directions from the East: I-82 – Exit 34, East Nob Hill Blvd, west bound.
Continue over the bridge over the railroad tracks – becomes West Nob Hill Blvd. –
Continue to 16th Ave and turn right (northbound) on 16th.
Continue 4 blocks to Tieton Drive and Turn left, (westbound) onto Tieton.
Hospital is on the right after S. 28th Avenue at 2811 Tieton Drive.



1 **16th Ave Station**
Memorial Occupational Medicine
Memorial Orthopedic Medicine
Memorial Interventional Pain Care
Memorial Rehabilitation and Physical Medicine
Memorial Therapy Services
1470 N. 16th Ave, 574-3300

2 **Business Services**
3803 W. Nob Hill Blvd, 575-8255

3 **Children's Village**
3801 Kern Rd, 574-3200

4 **Community Education**
2506 W. Nob Hill Blvd, 575-8484

5 **Cornerstone Medical Clinic**
402 S. 12th Ave, 248-3263

6 **Family Medicine of Yakima**
504 N. 40th Ave, 966-9480

7 **Garden Village**
206 S. 10th Ave, 453-4854

8 **Heritage Grove**
115 N. 10th St, 248-4173

9 **Home Health & Hospice**
1019 S. 40th Ave, 574-3600

10 **Maternal Health Services**
2903 W. Walnut Ave, 575-8160

11 **The Memorial Foundation**
2701 Tieton Dr, 576-5794

12 **Yakima Valley Memorial Hospital**
2811 Tieton Dr, 575-8000

13 **North Star Lodge/Memorial's Cancer Center**
808 N. 39t Ave, 574-3400

14 **`Ohana, Memorial's Mammography Center**
1515 W. Yakima Ave, 574-3860

15 **Pacific Crest Family Medicine**
311 S. 72nd Ave, 972-1818

16 **Sleep Center at Memorial**
406 S. 30th Ave, Suite 206, 452-5378

17 **Valley Imaging**
314 S. 11th Ave, Suite B, 248-7380

18 **Yakima Gastroenterology Associates**
3090 Creekside Loop, Suite 110, 248-6616

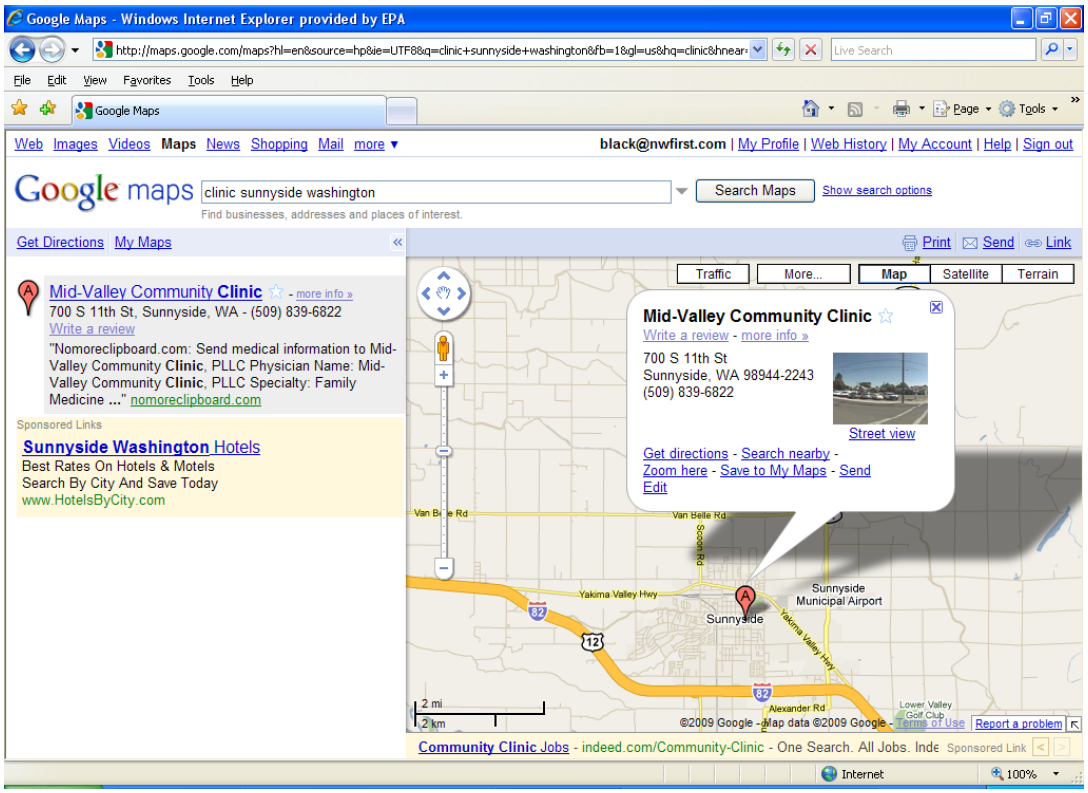
19 **West Pavilion I**
Cascade Surgical Partners, 575-3946
Yakima Neurosurgery Associates, 249-5217
Yakima Plastic Surgery Associates, 575-8633
Surgi-Center at Memorial, 576-0123
3003 Tieton Drive

Produced by MedMaps® 6/08 www.medmaps.com

700 South 11th Street
Sunnyside, WA 98944-2243

Get Directions
(509) 839-6822

Directions from the west: I-82, exit 67, Go north of South 1st Street, Right onto E. Lincoln Ave., Left onto S 11th Street. Hospital is 5 blocks north on the left side



4.0 Sampling Locations

Phase 2 results have focused the attention of this study on specific potential sources. In Phase 2 EPA teams were able to visit 337 homes and found more than 20% of them to exceed the MCL for nitrate. We also found 8 of those homes to have Coliform and/or E. coli bacteria. Based on our findings in Phase 2, we are now focusing on revisiting homes which significantly exceeded the MCL for nitrate. We plan to visit specific parcels located in areas of high concentrations of specific crop use, rural-residential septic systems, or down-gradient of dairies or feedlots. Phase 2 data were used to identify the locations of interest.

Property owners will have been contacted by the EPA Region 10 Outreach personnel or local cooperating contact personnel. The households chosen for Phase 3 follow-up sampling have helped previously in Phase 2. For the septic system characterization, 3 treatment plants will be contacted. For the crop-land survey, 3 crops with two fields of each crop will be selected for multi-increment sampling. For the AFO survey, 1 solitary dairy and one dairy complex which was identified in Phase 2 as being of particular interest will be visited. Their lagoons, manure piles, up and down-gradient wells and any onsite wells will be sampled.

Three types of samples will be collected during Phase 3:

- Water Wells (either domestic or dairy supply wells (20 to 23 bottles per location). Locations are identified as WW01 through WW29
- Liquids from Lagoons or Sewer Plant Influent Streams (16 bottles per location). Locations are identified as LG01 to LG15 and SP01 to SP03

- Soils/Solids from manure stockpiles or cropland (5 bottles per location). Locations are identified as SO01 to SO16

Table 6 Sampling Containers by Sample Type

Water Wells WW01 through WW29

☐ **WW** **"A-**MEL NH4-NO3+NO2-P-TKN"= **1-1L CU**. **"B-**MELAnions+ALK"= **1-1L CU**
"C-MEL Metals"= **1-1L CU** "**D-**VAL NO3" **1-1L CU**
"**E-**MEL Micro"= **2-100ml-Bact**, "**F-**ADA EST"= **1-2L-Pyrex Media**
"**G**-ADA Perc" **1-120ml-Plast**., "**H**-UNL NO3/NH4-N15"=**2-1L-Plastic**
(20) or "**I**-USGS WO 4433"= **1-1L Amber Glass** "**J**-UNL S.HOR"= **1-250ml Amb. Wide**
(23) w. gas "**K**-UNL Vet. P"= **1-250ml Amb. Wide** "**L**-UNL MWP"= **1-250ml Amb. Wide**
or pest dupe "**M**-UNL Extra"= **1-250ml Amb. Wide** "**N**-MEL Pest"= **3-60ml VOA**
"**O**-USGS SF6"= **2-1L Clear GAS**

Gas Study at this well? Yes No **if yes, take "P-**USGS Gas Study**"=3-gas study bottles – NOTE**

Record container numbers: 10Y-0 10Y-0 10Y-0

Pest Matrix-Dupe at this well? Yes No (check **Sample Count** sheet) **if yes, take another** "N-MEL Pest"= **3-60ml VOA**

Soils/Solids SO01 to SO17

☐ **SO**	"**Q**-MEL Pest"= **1-8oz Soil**	"**J**-UNL S.HOR"=**1-250ml Amb. Wide**
	"**K**-UNL Vet. P"=**1-250ml Amb. Wide**	"**L**-UNL MWP"=**1-250ml Amb. Wide**
(6)	"**M**-UNL Extra"=**1-250ml Amb. Wide**	**" U**-CAS-Extractable NO3/NH4 – Tot.N"-**1-16oz Soil**

Liquids from Lagoons or Sewer Plants LG01 to LG15 and SP01 to SP03

☐**LG/SP**	**"A-**MEL NH4-NO3+NO2-P-TKN"= **1-1L CU**.	**"B**-MELAnions+ALK"= **1-1L CU**
	"C-MEL Metals"= **1-1L CU**	"**E**-MEL Micro"= **2-100ml-Bact**,
	"**R**-ADA EST"= **1-1L-Pyrex Media**	"**H**-UNL NO3/NH4-N15"=**2-1L-Plastic**
(16)	"**I**-USGS WO 4433"= **1-1L Amber Glass**	"**J**-UNL S.HOR"= **1-250ml Amb. Wide**
	"**K**-UNL Vet. P"= **1-250ml Amb. Wide**	"**L**-UNL MWP"= **1-250ml Amb. Wide**
	"**M**-UNL Extra"= **1-250ml Amb. Wide**	"**N**-MEL Pest"= **3-60ml VOA**

4.1 Sample Collection Activities – Overview

Refer to the following 3 procedures for each of the 3 types of samples which will be collected in this field effort.

4.1.1 Water Well Samples from Taps or Spigots

a) Each week, each sample team will be provided a block of sample numbers as supplied by the lab for their use. For Phase 3 sampling we will use Scribe to print bottle labels and custody sheets for the several shipments we will generate per day. Determine the sample you are collecting (such as WW05 for Water Well 05 and examine the sheet for that sample. Peal off the **REG10 DATA BOOK** label from the sheet of labels for WW05 and place it on the field data form. Record the Lab Sample number on the field data sheet (eg. 10154205)

b) Samples will be collected by teams, each team composed of 2 or 3 EPA Region 10 field workers. A credentialed inspector will lead each sampling team.

c) Unfiltered samples will be collected directly from the disinfected faucet or other sampling point. Disinfect the spigot or faucet with the reagent grade alcohol provided in the spray bottle. As spring will be underway, sampling from an outdoor hose bib is acceptable.

d) The team will be setting up a flow-through cell for the collection of indicator parameters using a multi-parameter probe. Parameters such as dissolved oxygen, oxidation/reduction potential, conductivity, pH, and temperature will be recorded. The multi-parameter probe is calibrated each day prior to use in the field. Records from this calibration are kept in booklets dedicated to each instrument. These parameters are only collected from water wells – the instruments are not to be used in wastewater or lagoons.

e) Samples to be collected are included in Table 20 where the numbers of containers, the order of filling, the preservation and much other data useful to the sampling crew is tabulated. Please examine this table to determine if the

sample you are collecting needs a matrix spike duplicate for MEL Pesticides (3 extra 60ml VOAs) or if a dissolved Gas Study sample is needed here. This is shown by the shaded boxes on the **SAMPLE COUNT** sheet. The preprinted labels should have the correct bottles for each sample, but please check. Each preprinted sample label has the preservation type listed.

f) Before taking your first sample assemble the bottle set for this sample. The bottles labels should all have the same WW_ _ identifier. Check the count on the bottles – it should equal 20 (or 23) bottles. **Complete the bottle labels with the time and date and sampler initials.** Then place clear tape over each label. Then place the bottles in thin plastic bags to protect them and twist the bag around the neck of the bottle (no knots – it will be too difficult to remove after the bottle is filled appropriately.

g) For setup, a table may be temporarily placed near the sampling location. Any filter or aerator will be removed from the faucet prior to sampling. The faucet will be sprayed with reagent-grade ethanol as a disinfectant and the excess wiped away with a clean paper towel. A flow-through cell will be plumbed into the system with tubing and various sizes and combinations of adapters. The discharge from the flow-through cell will be directed into the sink or to the ground if outside.

h) The team will make certain that no water softening equipment or reverse-osmosis treatment units are in use. If such equipment is present, the sample will be obtained before (upstream) of the softening or RO unit. Filtered samples will not be collected.

i) The selected household cold water faucets/taps will be purged for a minimum of 10 minutes prior to sampling. The purpose of purging is to clear the delivery system of stagnant water and provide fresh water for sampling. Ideally, the sampling team will hear the pressure tank cycle at least twice during the purging. Often you can see and hear the delivery pressure cycle at the tap.

j) All field parameter values will be monitored and recorded at 1 minute intervals during purging, and temperature, DO and oxidation-reduction potential stabilization will be used as primary indicators that the system has been adequately purged and that sampling may begin. In some cases, the parameters will not stabilize within a reasonable period of time. Fluctuation is usually caused by the normal cycling of the well pump as controlled by the pressure tank switch. After 10 minutes, experienced field personnel will determine when sufficient water has been purged in these cases.

k) After 10 minutes of purging, take a nitrate test strip and a Ferrous Iron measurement. Record the values on the field data sheet.

l) After the purge is compete, the hose to the flow-through cell will be disconnected from the tap and the bottles filled without turning off the flow of water (the flow rate may be decreased if desired for ease in filling. Put on gloves if you haven't yet.

m) Fill the entire bottle set (20 or 23 bottles) for this location. The bottles are to be filled in the order specified on the field data sheet. Hold the caps in one hand and the bottle in the other – do not place the caps on any surface. Screw the caps securely onto each bottle after it is filled. Note the time of sampling on the field data form.

n) For the SF6 sample, without turning off the water, reconnect the discharge piping to the tap and place a smaller tube into the discharge piping and direct the tube into the glass container and allow the water to enter from the bottom and flow out the opening. Allow the water to overflow for at least 3 volumes from this container, then slowly remove the tubing and cap without any trapped gas bubbles (like a VOA). Tape the cap in place with vinyl/electrical tape in the direction so that the tension of the tape tends to screw the cap closed

o) IF you are filling a dissolved gas study sample, fill the containers (3) as shown in the training completely under water in a plastic dish tub with the water actively overflowing the tub as you work the cap into the container with the hypodermic needle allowing the water to exit as the seal is seated into the container.

p) Some samples require preservation. Container "A-MEL NH4-NO3+NO2-P-TKN" needs addition of H2SO4 (sulfuric acid) to pH less than 2.

q) Container "C-MEL Metals" needs preservation with HNO3 (nitric acid) to pH less than 2.

r) Sample pH will be spot-checked in the field to determine adequate preservation, and additional acid will be added if needed. Sample preservation requirements are included in Table 7. For the spot check, pour a small amount of the acidified sample into a clean 50 ml beaker and using a multi-indicator test strip, make sure the pH is less than 2. If so, cap the bottle, if not add more acid and recheck.

s) Samples will be placed in coolers for shipment or delivery to each designated laboratory. Please refer to the Sample Container SOP for more details. Samples will be shipped as needed to meet analytical holding times. Please note that SF6 samples and gas studies are not iced – treat each sample type appropriately

4.1.2 Solid Samples – either Manure or Agricultural Fields

a) Each week, each sample team will be provided a block of sample numbers as supplied by the lab for their use. For Phase 3 sampling we will use Scribe to print bottle labels and custody sheets for the several shipments we will generate per day. Determine the sample you are collecting (such as SO05 for Soil Sample 05 and examine the sheet for that sample. Peal off the **REG10 DATA BOOK** label from the sheet of labels for SO05 and place it on the field data form. Record the Lab Sample number on the field data sheet

b) Samples will be collected by teams, each team composed of 2 or 3 EPA Region 10 field workers.

c) After becoming familiar with the quantity or area to be representatively sampled plan a route through the area or around the pile so that you can systematically collect 40 subsamples for compositing. We are collecting multi-increment samples in the interest of decreasing the variability we anticipate from sampling a heterogeneous material like a manure pile or an agricultural field. There is no need to climb onto a pile or to attempt to "enter" the pile – we will work from the outer edges and call our work done. People have died sampling manure piles – methane gas, hydrogen sulfide and ammonia are all on the list of inhalation hazards. We will outfit the sampler with a O2 and combustible gas meter to ensure the sampler stays in an atmosphere conducive to health. The meter will alarm if O2 falls below 19.5%.

d) Assemble the bottle set of 5 bottles for the Soil/Solid sample. Label the containers if they are not yet labeled. Add the date and time and your initials to the label and cover with transparent tape. Put on new gloves.

e) For sampling the agricultural field, you might plan a diagonal course through the field attempting to reach at least 1/3 of the way across. You could head for the center pivot point if the field has one. When you get to 20 of 40 subsamples, turn 90 degrees and you should re-intersect the boundary of the field at your last sample. For example – you select a direction across the field and plan on collecting a small sub-sample every 25 steps. Where ever your 25th footstep lands, take your sampler (prepared spoon or syringe) and insert it at the ball of your foot.

f) Try to recover about 1 teaspoon or so of soil centered about 1-inch below the surface. Not too deep and not just at the surface. Place the soil into the prepared stainless steel bowl. Take 25 more steps and repeat until you have collected 40 subsamples.

g) If it is a furrowed field you should have a mix of furrows and ridges. Please systematically deviate so as to not step on the farmers crop – if your foot will hit a plant, just systematically move left or right each time. You should have about 1 liter of soil in the bowl. Not much more or the homogenizing step will be incomplete and a lot of work.

h) Return to the area with the sample containers and work to homogenize the sample. The laboratory will only pull 5 grams off the top, so we want every bit of the jar to be representative of the field you just traversed. We are looking for trace organic compounds. Everything that has contacted the sample has been baked in an oven at 450 degrees C. for 4 hours. These bottles are completely organic free. The only thing that should contact the soil is the baked spoon, the dedicated syringe, the baked bowl and the baked sample containers.

i) Fill the containers only 70% full so they don't burst when frozen at -20 degrees prior to shipment.

j) Check you container count against the list on the field data sheet – 5 Containers made up of 1-8oz soil and 4-250ml wide ambers Check that the sample number matches on each bottle and matches the data sheet.

k) All these soils will be frozen prior to shipment so they can be bagged and go directly into an ice chest with ice.

4.1.3 Lagoon Sampling

a) Each week, each sample team will be provided a block of sample numbers as supplied by the lab for their use. For Phase 3 sampling we will use Scribe to print bottle labels and custody sheets for the several shipments we will generate per day. Determine the sample you are collecting (such as LG15 for Lagoon Sample 15 or SP02 for Sewer Plant 02) and examine the field data sheet for that sample type. Peal off the **REG10 DATA BOOK** label from the sheet of labels for LG15 and place it on the field data form. Record the Lab Sample number on the field data sheet.

b) Samples will be collected by teams, each team composed of 2 or 3 EPA Region 10 field workers. Specialized safety equipment is necessary for lagoon sampling. We will have lifejackets available and a rope on the bank while sampling.

c) We want to sample different parts of the lagoon system. One target will be the "freshest or youngest" end of the system and the other will be the lagoon just before the liquids are removed by pumping onto agricultural fields. By collecting both, we can gain additional data on how the waste changes with passage through the system as well as how sensitive to degradation some of the compounds are during their residence.

d) After becoming familiar with the lagoon system select your sampling location. If there is any question as to the stability of the bank or you ability to safely remove these liquids, rope up and/or put on the lifejacket. People have died collecting lagoon samples. The lagoon environment may be full of hydrogen sulfide, ammonia and other compounds at concentrations immediately dangerous to life and health.

e) Before taking your first sample assemble the bottle set for this sample. The bottles labels should all have the same LG_ _ or SP _ _ identifier. Check the count on the bottles – it should equal 16 bottles. Bottle labels should have clear tape over them after the time and date and sampler initials have been written. Then place the bottles in thin plastic bags to protect them and twist the bag around the neck of the bottle (no knots – it will be too difficult to remove after the bottle is filled appropriately. As the teams gain experience with these activities they may modify this procedure to best accomplish the sampling. If it is found better to assemble the bottle set, then fill, then seal, then clean the bottles after sampling, then sterilize the outside, then label the containers at each location the teams should do so. With the exception of collecting duplicate

samples from a single lagoon, sampling should be completed and bottles labeled and packed before starting sampling for another location..

f) Most of the samples will be iced after filling and can be filled to 90%. However, the **Two-1-liter Plastic** bottles and the **Four -250 ml wide amber** bottles are **only filled 70% full so the may be frozen at -20 F. for shipment.**

g) We are using a telescoping pole to hold a dedicated bottle which will be used to remove the liquids from the lagoon. The dedicated 1-liter small mouth bottle has been baked at 450 degrees C. to remove any trace-organic compounds.

h) Both samplers should put on new gloves.

i) Use the telescoping pole to break through any crust that may be on the lagoon near where you are working. When you have an area open, lower the bottle through the hole and attempt to reach as far as the pole will safely allow. We are attempting to integrate our sample across the accessible depth of the lagoon. As the bottle fills, move the bottle across this depth range to provide as representative a sample as possible.

j) When the bottle stops bubbling, bring it to the surface and prepare to pour the contents into the containers making up the bottle set. The second sampler should open the containers and working so as to minimize splashing and the production of aerosols, transfer the contents of the 1-liter container to each of the containers in the order they are presented on the field data sheet.

k) As each bottle is filled to the desired 70% or 90% depending on type, firmly screw on the caps and wipe down the top with a paper towel. Remove the plastic bag, wipe down the bottle again with the antibacterial liquid provided and place the container in a new plastic bag.

l) When the bottle set is complete, re-tighten each lid, count all the containers and check that all the sample numbers are correct.

m) Rinse the sampler in a bucket of water. Dispose of the now contaminated 1-liter transfer bottle, then rinse the sampler with the antibacterial liquid.

n) Carefully pack the containers in bags and on ice (all the containers for lagoon and sewer plant samples can be iced, later 5 of them will be frozen as well).

4.2 Sample Shipments

Shipping for all analytes will be by UPS or courier –last shipment time is 6:00pm

Shipment will use Account #973856 User ID - USEPAR10 Password - 4U2USE

UPS Customer Center
STAFFED LOCATION
501 W VALLEY MALL BLVD
UNION GAP, WA 98903
Tel.: 800-742-5877

Open 1pm to 6pm – M- F
Driving Directions off I-82

At exit 36, take ramp right for Valley Mall Blvd. toward Union Gap	0.3 mi
Turn left onto E Valley Mall Blvd	0.4 mi
Keep left onto W Valley Mall Blvd	0.1 mi
Arrive at W Valley Mall Blvd	

Shipping Locations and Requirements

Analyte/Analysis	Location	Contacts	Comments
Pesticides and General Chemistry	Manchester Environmental Lab 7411 Beach Drive East Port Orchard, WA 98366	Karen Norton, ESAT Contractor; She will be receiving the samples 1-360-871-8760	3 cubitainers and 3 VOAs and or 8oz soil jars
Nitrate, Bacterial and soil total-nitrogen and extractable nitrate and ammonia	Cascade Analytical 1008 West Ahtanum Road Union Gap, WA 98903-1897	Andy Schut ("Skut") (509) 452-7707	Hand carry. We will use the lab for April 12th-13th and April 18th-April 22nd for microbial samples. Whole period on nitrate 300 samples.
Estrogen Hormones (Ada EST) And Perchlorate	USEPA KERR Lab 919 Kerr Research Drive Ada, OK 74820 1-580-436-8563 (580)436-8920	Hutchins.steve@epa.gov Greenwood.andrew@epa.gov Bennett.shauna@epa.gov	Include a completed "Analytical Sample Record" form with each Ada shipment. Email contacts when shipping. No Friday, Saturday, or Sunday shipments
Isotopic (UNL NO3/NH4-N15) Veterinary antibiotics, (UNL VP) Mixed wastewater pharmaceuticals (UNL MWP) Steroid hormones (UNL SH),	Daniel Snow, Laboratory Director Water Center School of Natural Resources 202 Water Sciences Lab University of Nebraska Lincoln, NE 68583-0844	Dsnow1@uni.edu phone (402) 472-7539 fax (402) 472-9599	Notify Dr. Snow by email when shipping samples Ship frozen at -20 with ice
Trace organics (USGS WO)	Gary Cottrell, Supervisory Chemist NWQL National Water Quality Lab Building 95, Ent E-3 Denver Federal Center Denver, CO 80225-0046	cottrell@usgs.gov phone (303) 236-3490 fax (303) 236-3499	Keep at <4 degrees C. Ship daily except Friday, Saturday, and Sunday

Analyte/Analysis	Location	Contacts	Comments
Age Dating USGS SF6 and Gas Study (AR/N)	USGS CFC laboratory 432 National Center 12201 Sunrise Valley Dr. Reston, VA 20192	Peggy Widman pkwidman@usgs.gov (703) 648-5847 703-648-5347	**Do not chill –** **NO ICE** Keep at room temp until shipped at room temp Email sample spreadsheet

4.3 Sample Collection Equipment

Suggested Sample Equipment for Yakima Valley Phase 3 Field Activities

General	Safety	Emergency
Copy of this QAPP	Water Proof (Rubber) Boots	Phone numbers (back page of Field Data Books)
EPA ID and/or Credentials	EPA Identity Wear	Cell Phone
Field Data Books and Sample Labels	Leather and Rubber gloves	Map Books
Portable Table	Soap, towels, and water for washing hands	911
Camera	Sprayer for anti-bacterial solution and 3 tubs for boot wash	
Waterproof Pens & Markers	Acid cleanup kit (sodium bicarbonate in Ziploc)	
Clipboard 1 long for custody paperwork/team	Eye protection	
Fashlight / LED Headlamp	Hand Sanitizer	
Sample containers	Chapstick	
Ice Chest plus extras for equipment transport	Sunglasses	
Table of Homes to be Sampled	First Aid Kit	
GPS for each team	Brushes for boots and equipment	
Flow-Through Cell	Spray Bottles and Verikon	
Multi-parameter Probe	TomTom Navigation Systems	
Plumbing / Connections and clamps	Neosporin to keep hands skin from cracking	
Calibration Solutions	Positive attitude in community interactions	
Custody Sheets/Custody Seals/blank labels	Situational Awareness	
Shipping Forms – Duct and Fiber Tape	Rain gear	
Forms for each Laboratory Receiving Samples	Trash Bags	
Wash Tub for Dissolved Gas Study Sample filling	food and drinks	
Nitric Acid kits for Metals Sample Preservation		

General	Safety	Emergency
Sulfuric Acid kits for MEL Cubitainer for TKN+		
pH Indicator Strips		
Nitrate Indicator Strips		
Ferrous Iron Test Kit		
Telescoping Sampling Poles		
Write in the Rain books		
Clear tape for covering labels		
Field Ice for samples		
Bubble Wrap		
Extra tubing for SF6 sample filling		

All sample jars used for this project will be new and clean. All containers have been prepared in advance as directed by the various labs performing analyses for this project.

4.4 Decontamination Procedures

4.4.1 For drinking water in private homes.

This medium is expected to be considerably safer than much of what we typically encounter in environmental sampling. Still, keep the work area as clean as possible; minimize contact with the water during purging and filling the bottles and use hand sanitizer upon arriving and departing each location.

4.4.2 For AFOs:

This activity has the potential to both expose you as a sampler to pathogens and chemicals of concern, but also to transmit foreign pathogens to the facility. In light of this we will be decontaminating equipment during the demobilization step from each facility. The decontamination procedure will include a standard contamination reduction corridor with at least two stations set up to remove gross contamination, then sterilize the protective clothing and equipment, then rinse the protective clothing and equipment, and then package material for transport to the next sampling station.

4.4.3 For Cropland Soil Sampling

After completing the collection of each crop land multi-increment sample, the sampler will pass through the contamination reduction corridor setup for the facility being sampled. If the field being sampled does not receive manure or liquid effluent from a dairy, then boots can be removed and packed into plastic bags for the next use.

4.5 Sample Handling and Custody Requirements

This subsection describes sample identification and chain-of-custody procedures that will be used for the Phase 3 Yakima Basin Nitrate Study field activities. The purpose of these procedures is to ensure that the quality of the samples is maintained during collection, transportation, storage, and analysis. All chain-of-custody requirements comply with USEPA Manchester Environmental Laboratory's (MEL's) SOPs for sample handling. All sample control and chain-of-custody procedures will follow MEL's SOPs or the *Contract Laboratory Program Guidance for Field Samplers* (EPA 2004b).

Examples of sample documents used for custody purposes will be provided to the team and include the following:

- Custody seals,
- Chain-of-custody records or traffic reports, and
- Field logbooks with data forms bound for each team.

During the field effort, the site manager or delegate is responsible for maintaining an inventory of these sample documents. This inventory will be recorded in a cross-referenced matrix of the following:

- Sample location,
- Sample identification number,
- Analyses requested and request form numbers,
- Chain-of-custody record numbers,
- Bottle lot numbers, and
- Air bill numbers.

Brief descriptions of the major sample identification and documentation records and forms are provided below.

4.6 Sample Identification

All samples will be identified using the sample numbers assigned by the EPA RSCC. Each sample label will be affixed to the jar and covered with clear tape. A sample tracking record will be kept as each sample is collected. The following will be recorded: location, matrix, sample number, observations, and depth. In addition to the EPA-assigned sample number, samples will be tracked with a sample code system designed to allow easy reference to the sample's origin and type. The sample code key is the first part of each location name. The second part is the 8-digit RSCC issued lab-sample number.

4.7 Sample Labels

To minimize handling of sample containers, labels will be completed before sample collection. After being attached firmly to the sample container, the labels will completed with the date and time of sampling and the sampler's initials. The labels will then be protected with clear tape. The sample labels will provide the following information:

- Sample location and number;
- Date and time of collection,
- Sampler initials,
- Analyses required, and
- Preservation (when required).

4.8 Custody Seals

Custody seals are preprinted gel-type seals, designed to break into small pieces if the seals are disturbed. Sample shipping containers (coolers, drums, cardboard boxes) will be sealed in as many places as necessary to ensure security. Seals will be signed and dated before use. Clear tape will be placed over the seals to ensure that the seals are not broken accidentally during shipment. Upon receipt at the laboratory, the custodian will

check (and certify by completing the package receipt log) that seals on shipping containers are intact.

4.9 Chain-of-Custody Records and Traffic Reports

For samples to be analyzed at the EPA MEL the chain-of-custody records, analyses-required forms, and/or analytical traffic report forms will be completed as described in the *Contract Laboratory Program Guidance for Field Samplers* (EPA 2004b). The chain-of-custody record, analyses-required forms, and analytical traffic reports will be completed fully at least in duplicate by the field technician designated by the site manager as responsible for sample shipment to the appropriate laboratory. Information specified on the chain-of-custody record will contain the same level of detail found in the site logbook, except that the on-site measurement data will not be recorded. The custody record will include the following information:

- Name and company or organization of person collecting the samples,
- Date samples were collected,
- Type of sampling conducted (composite or grab),
- Sample number (using those assigned by the EPA RSCC),
- Location of sampling station
- Number and type of containers shipped,
- Analysis requested, and
- Signature of the person relinquishing samples to the transporter, with the date and time of transfer noted and signature of the designated sample custodian at the receiving facility.

If samples require rapid laboratory turnaround, the person completing the chain-of-custody record(s) will note these or similar constraints in the remarks section of the custody record.

The relinquishing individual will record all shipping data (e.g., air bill number, organization, time, and date) on the original custody record, which will be transported with the samples to the laboratory and retained in the laboratory's file. Original and duplicate custody records, together with the air bill(s) or delivery note(s), constitute a complete custody record. It is the site manager's responsibility to ensure that all records are consistent and that they become part of the permanent job file.

4.10 Field Logbooks and Data Forms

Field logbooks of data forms are necessary to document daily activities and observations. Bound logbooks of data forms have been assembled for each sampling team for the project. Documentation will be sufficient to enable participants to reconstruct events that occurred during the project accurately and objectively at a later time. All daily logs will be kept in a bound notebook. All entries will be made in waterproof ink, dated, and signed. No pages will be removed for any reason.

If corrections are necessary, these corrections will be made by drawing a single line through the original entry (so that the original entry is legible) and writing the corrected entry alongside. The correction will be initialed and dated. Corrected errors may require a footnote explaining the correction.

4.11 Photographs

Photographs will be taken as directed by the team leader. Documentation of a photograph is crucial to its validity as a representation of an existing situation. The following information will be noted in the project or task log concerning photographs:

- Date and time,
- Photographer (initials),
- Description of photograph taken,
- Sequential number of the photograph,
- Camera lens system used, and
- Direction.

4.12 Custody Procedures

The primary objective of chain-of-custody procedures is to provide an accurate written or computerized record that can be used to trace the possession and handling of a sample from collection to completion of all required analyses. A sample is in custody when it is:

- In someone's physical possession,
- In someone's view,
- Locked up, or
- Kept in a secured area that is restricted to authorized personnel.

4.13 Field Custody Procedures

The following guidance will be used to ensure proper control of samples in the field:

- As few people as possible will handle samples.
- Coolers or boxes containing cleaned bottles will be sealed with a custody tape seal during transport to the field or while in storage before use. Sample bottles from unsealed coolers or boxes, or bottles that appear to have been tampered with, will not be used.
- The sample collector will be responsible for the care and custody of collected samples until they are transferred to another person or dispatched properly under chain-of-custody rules.
- The sample collector will record sample data in the field logbook.
- The site team leader will determine whether proper custody procedures were followed during the field work and will decide if additional samples are required.

When transferring custody (for example, releasing samples to a shipping agent), the following will apply:

- The coolers in which the samples are packed will be sealed and accompanied by two copies of the chain-of-custody record(s). When transferring samples, the individuals relinquishing and receiving them must sign, date, and note the time on each of the chain-of-custody record(s). This will document sample custody transfer.

- Samples will be dispatched to the laboratory for analysis with separate chain-of-custody records accompanying each shipment. The chain-of-custody records will be signed by the relinquishing individual, and the method of shipment, name of courier, and other pertinent information will be entered in the chain-of-custody record before placement in the shipping container. Shipping containers will be sealed with custody seals for shipment to the laboratory.
- All shipments will be accompanied by chain-of-custody records identifying their contents. The original custody records kept in a zip-lock bag and taped inside the lid of the cooler will accompany each cooler shipment. The other copies will be distributed appropriately to the site team leader and site manager.
- If sent by common carrier, a bill of lading will be used. Freight bills and bills of lading will be retained as part of the permanent documentation.

4.14 Laboratory Custody Procedures

A designated sample custodian at the laboratory will accept custody of the shipped samples from the carrier and enter preliminary information about the package into a package or sample receipt log, including the initials of the person delivering the package and the status of the custody seals on the coolers (for example, broken versus unbroken). The custodian responsible for sample log-in will follow the laboratory's SOP for opening the package, checking the contents, and verifying that the information on the chain-of-custody agrees with the information on the samples received. The commercial laboratory will follow its internal chain-of-custody procedures as stated in the laboratory QA manual. The laboratory will check the temperature blank inside the cooler and document it in the sample log-in form. Should the temperature be greater than what is required by the Statement of Work or the method, the sample custodian will inform the region and proceed to follow the course of actions stipulated in the SOW or specified by the regional QA Coordinator.

4.15 Analytical Methods Requirements

Please Refer to Table 4 for a list of requested analytes, methods, containers, preservation methods and holding times. None of the samples in this project need be retained by the lab after analysis. These samples are either from drinking water wells and as such are considered non-hazardous or are biological materials the potential for bacterial or viral contamination.

5.0 Quality Assurance & Quality Control

Components of quality assurance and quality control will include data quality objectives, equipment calibration, analytical procedures and reporting levels, quality control procedures, data reduction, validation, and reporting, performance and systems audits, data assessment, corrective action, and quality assurance reports. Manchester will follow its written QA/QC Plan to assure data quality.

Quality control in the field shall include field instrument checking and re-calibration, if necessary, immediately prior to the beginning of sample collection each morning.

Duplicate samples have been incorporated into the sampling design of the project. Duplicate frequencies range from 100% for the SF6 samples, to 1 in 20 for the matrix

spike dupes for pesticides. Most of the samples for this project are duplicated at a rate of 1 in 10 for field duplicates.. In addition, approximately 5% of the samples will be analyzed in duplicate (analytical duplicate). If the method will allow, a matrix spike and matrix spike duplicate will also be analyzed per batch of 20 samples. The spike recoveries and the relative percent differences (RPDs) are calculated and reported for each parameter. The required precision and accuracy for this project is 20% relative percent difference (RPD) and accuracy, as measured by recovery of spikes is reported in the laboratory specific tables and is usually 80-120% but varies with matrix and analysis.

Transfer blanks will be incorporated into the sample schedule at a rate of approximately 5% or once per sample location type. Transfer blanks will consist of sterile, analyte free water which will be poured from a laboratory supplied storage container into a designated sample container during the regular collection of field samples while on-site. The data generated from these blanks will be used to establish a set of empirical data supporting the adequacy of the sample collection and analytical processes in minimizing the contamination of samples. Transfer blanks will be used primarily for the water samples submitted to MEL in addition to nitrate and coliform drinking water analyses sampled from domestic groundwater wells.

5.1 Instrument Calibration and Frequency

All field instruments will be calibrated each day prior to use using standards traceable to NBS or using procedures relying on physical constants (such as dissolved oxygen at equilibrium with distilled water at a known temperature). Lab instruments are under a rigorous program of QA and which will be followed under Manchester Lab standard protocols. Field instruments measure a wide range of environmental variables and those measurements are subject to error to the extent that field personnel are unfamiliar with the means used to obtain the measurement. To address this Appendix 1 contains specific information on each of the parameters we will measure, what each measurement means and detailed descriptions of how to calibrate for each parameter.

5.2 Inspection/Acceptance Requirements for Supplies and Consumables

Sample bottles used for microbial testing will be appropriately cleaned and sterilized as per MEL SOP MiG001A. They will be certified clean polypropylene bottles (250 or 500 mL). All sample jars or cubitainers used for chemical analysis in this project will be new and certified clean provided by the laboratory. Field personnel will make note of the information on the certificate of analysis that accompanies sample jars to ensure that they meet the specifications and guidance for contaminant free sample containers. All containers used for trace organic or hormone or veterinary pharmaceutical analyses have been baked at 450 degrees C for at least 4 hours to be free from contamination. All sampling equipment used in the compositing for these analyses have also been baked in the same way.

5.3 Data Management

Field Data Sheets, photos, GPS location data and the Sample Labels and Chain of Custody Data Sheets will be used to document the sampling and inspection activities. For each sample location, the following will be recorded on a dedicated data sheet for each location as shown below:

Yakima Nitrate Study – Phase 3 – Field Data Form

Database Y

Sample Code (Circle) WW SO LG SP +No._____ ____ Date_____

Lab Sample number _____ U#_____ Credentials Shown on Entry? Y N

Sample Type: (matches container label & COC)

☐ **WW** **"A-**MEL NH4-NO3+NO2-P-TKN"**= 1-1L CU**. **"**MEL B-Anions+ALK"**= 1-1L CU** **"C-**MEL Metals"**= 1-1L CU** "**D**-VAL NO3" **1-1L CU**
"**E**-MEL Micro"**= 2-100ml-Bact**, "**F**-ADA EST"**= 1-2L-Pyrex Media** "**G**-ADA Perc" **1-120ml-Plast**., "**H**-UNL NO3/NH4-N15"**=2-1L-Plastic**
(20) or **"**I-USGS WO 4433"**= 1-1L Amber Glass** "**J**-UNL S.HOR"**= 1-250ml Amb. Wide** "**K**-UNL Vet. P"**= 1-250ml Amb. Wide**
(23) w. gas "**L**-UNL MWP"**= 1-250ml Amb. Wide** "**M**-UNL Extra"**= 1-250ml Amb. Wide** "**N**-MEL Pest"**= 3-60ml VOA**
or pest dupe "**O**-USGS SF6"**= 2-1L Clear GAS**
Gas Study at this well? Yes No **if yes, take "P**-USGS Gas Study**"=3-gas study bottles – NOTE # 10Y-0_____ 10Y-0_____ 10Y-0_____**
Pest Matrix-Dupe at this well? Yes No (check **Sample Count** sheet) **if yes, take a** "**T**-MEL Pest"**= 3-60ml VOA**

☐ **SO** "**Q**-MEL Pest**=1-8oz Soil** "**J**-UNL S.HOR" **=1-250ml Amb. Wide** "K-UNL Vet. **P**"**= 1-250ml Amb. Wide**
(6) "**L**-UNL MWP"**= 1-250ml Amb. Wide** "**M**UNL Extra"**=1-250ml Amb. Wide**
"U-CAS-Extractable NO3/NH4 – Tot.N"-**1-16oz Soil**

☐**LG/SP** **"A-**MEL NH4-NO3+NO2-P-TKN"**= 1-1L CU**. **"B-**Mel-Anions+ALK"**= 1-1L CU** **"C-**MEL Metals"**= 1-1L CU**
"**E**-MEL Micro"**= 2-100ml-Bact**, "**R**-ADA EST"**= 1-1L-Pyrex Media** "**H**-UNL NO3/NH4-N15"**=2-1L-Plastic**
(16) "**I**-USGS WO 4433"**= 1-1L Amber Glass** "**J**-UNL S.HOR"**= 1-250ml Amb. Wide** "**K**-UNL Vet. P"**= 1-250ml Amb. Wide**
"**L**-UNL MWP"**= 1-250ml Amb. Wide** "**M**-UNL Extra"**= 1-250ml Amb. Wide** "**N**-MEL Pest"**= 3-60ml VOA**

Name/Address Same as Last Sheet?: No Yes (if yes, leave blank)

Family or Facility Name _____Address:_____

Phone:_____ City/Zip:_____

Water Softener or Reverse Osmosis Unit this well? YES / NO / NA(soil) Alternate Sampling Location Selected? YES / NO / NA
Describe Alternate Sampling Location for Collection before Treatment:
_____

GPS at sample location: Lat_____ Long_____Waypoint Number(s):__ 60 Sec. Avg? YES / NO
Time of Purge Start: _____ Caps off DO and PH probe? Yes / No

Minute 0: TEMP: PH: REDOX: COND: DO:
Minute 1: TEMP: PH: REDOX: COND: DO:
Minute 2: TEMP: PH: REDOX: COND: DO:
Minute 3: TEMP: PH: REDOX: COND: DO: place pre-printed datasheet sample label here
Minute 4: TEMP: PH: REDOX: COND: DO:
Minute 5: TEMP: PH: REDOX: COND: DO:
Minute 6: TEMP: PH: REDOX: COND: DO:
Minute 7: TEMP: PH: REDOX: COND: DO:
Minute 8: TEMP: PH: REDOX: COND: DO:
Minute 9: TEMP: PH: REDOX: COND: DO:
Time of Purge End:_____

Test Strip Reading from Nitrate field test (circle range) 0 <1 <2 <5 <10 <20 <50 50+ mg/l
Reading from Ferrous Iron field test (circle range) 0 <0.5 <1 <2 <3 <4 <5 <6 <7 <8 <9 <10 10+mg/l

Sample Collection Time:_____

Color/Odor and any notes on sample appearance or collection process:_____

Log of gas study vial numbers or any photos taken and description of subject (use back for more notes):
_____

Sampler:_____ Assisted by:_____

Clean equipment, replace caps on PH and O2 probe, count samples against lists above, d-con you, thank owner/operator

The Sample Label and Chain of Custody Data Sheets will have the following information:
* Sample type and Location # (WW07)
* Laboratory sample number (10154207)
* Date and Time of sample collection (added in the field by sample team)
* sampler's name or initials (added in the field by sample team)
* Analyses Requested
* Preservative

For fixed laboratory analyses, field duplicates will be assigned a separate unique sample identifier and will be submitted 'blind' to the analytical laboratory. This will include a field blank for all analyses except the SF6 and Isotopic analyses

All data generated during this project will be processed, stored, and distributed according to each laboratory's SOPs.

6.0 Assessment and Oversight

6.1 Assessments and Response Actions

The EPA Field Sampling Project Manager will be responsible for reviewing field log notebooks for accuracy and completeness within 24 hours of each inspection. Sample results provided to the Sampling Project Manager by the laboratory will be assembled for a sampling report. The EPA Field Sampling Project Manager will compare the sample information on the field data sheets with the analytical results to ensure that no transcriptions errors have occurred.

With the exception of the microbiological analyses, RPDs between field duplicate and analytical duplicate measurements will be calculated by the laboratory. RPD's greater than the project requirements will be noted in the associated inspection reports.

Laboratories routinely perform performance checks using different program specific quarterly blind and double blind check standards. Each method of analysis requires specific QA/QC runs that must be complied with by the laboratory performing the analysis. An internal assessment of the data and results are also routinely conducted by the appropriate supervisors and the Laboratory QA Coordinator. No additional audits will be performed on the laboratory for this project.

Corrective action procedures that might be implemented from QA results or detection of unacceptable data will be developed if required and documented in Attachment 2.

6.2 Reports to Management

Only the data validation reports with the properly qualified data shall be provided by the laboratory to the Field Sampling Project Manager. If, for any reason, the schedules or procedures above cannot be followed, the EPA Field Sampling Project Manager must complete the Attachment 1- Sample Alteration Form (SAF). The SAF should be

reviewed and approved by the QAO. The laboratory should be given a copy of the QAO approved SAF for reference and project file.

7.0 Data Validation and Usability

7.1 Data Review, Validation, and Verification Requirements

The criteria for the validation will follow those specified in this QA plan and the criteria specified in the methods.

7.2 Validation and Verification Methods

All data generated shall be validated in accordance with the QA/QC requirements specified in the methods, and the technical specifications outlined in the QAPP. The summary of all analytical results will be reported to the EPA Field Sampling Project Manager. The raw data for this project shall be maintained by the laboratory. Data validation will be performed by the laboratory for all the analyses prior to the release of data in accordance with each laboratory's quality assurance plan. The laboratory will also archive the analytical data into their laboratory data management system.

7.3 Reconciliation with User Requirements

All data and related information obtained during the course of this project will be included in a data report package. Non-detected nitrate results will be assessed to determine if they meet the reporting limit requirements set forth in the QAPP and the drinking water MCLGs.

8.0 Analytes, Methods, Holding Times and Preservation

Table 7 – Manchester Environmental Laboratory – General Chemistry

Analyte (9)	Method	Prep Method	Reporting Limit	Container type	Number of Containers	Container #	Bias (accuracy)	Variability (precision)	Hold time	preser
Ammonia	350.1		.05 or 0.3 mg/l	1-L poly	1	1	80-120%	+/- 20%	28 Days	4deg.C.-H2
Nitrate + Nitrite	353.2		0.05mg/l	1-L poly	1	1	80-120%	+/- 20%	28 Days	4deg.C.-H2
P - Total Phosphorus	365.1		0.02mg/l	1-L poly	1	1	80-120%	+/- 20%	28 Days	4deg.C.-H2
Kjeldahl Nitrogen	351.2		0.5mg/l	1-L poly	1	1	80-120%	+/- 20%	28 Days	4deg.C.-H2
Nitrate	300.0		0.04 to 0.3 mg/L	1-L poly	1	See Valley Laboratory	80-120%	+/- 20%	48 Hours	4de
Bromide	300.0		0.2mg/l	1-L poly	1	2	80-120%	+/- 20%	28 Days	4 de
Chloride	300.0		0.06mg/l	1-L poly	1	2	80-120%	+/- 20%	28 Days	4 de
Fluoride	300.0		0.04mg/l	1-L poly	1	2	80-120%	+/- 20%	28 Days	4 de
Sulfate	300.0		0.3mg/l	1-L poly	1	2	80-120%	+/- 20%	28 Days	4 de
Alkalinity	2320B		5.0 mg/l	1-L poly	1	2	80-120%	+/- 20%	14 Days	4 de
Analyte specifications for metals (16)										
As - Arsenic	200.7	200.2	45 ug/l	1-L poly	1	3	80-120%	+/- 20%	6 mo.	HNO3
Ba - Barium	200.7	200.2	1 ug/l	1-L poly	1	3	80-120%	+/- 20%	6 mo.	HNO3
Ca - Calcium	200.7	200.2	30ug/l	1-L poly	1	3	80-120%	+/- 20%	6 mo.	HNO3
Cd - Cadmium	200.7	200.2	3 ug/l	1-L poly	1	3	80-120%	+/- 20%	6 mo.	HNO3
Cr - Chromium	200.7	200.2	10 ug/l	1-L poly	1	3	80-120%	+/- 20%	6 mo.	HNO3
Cu - Copper	200.7	200.2	10 ug/l	1-L poly	1	3	80-120%	+/- 20%	6 mo.	HNO3
K - Potassium	200.7	200.2	700ug/l	1-L poly	1	3	80-120%	+/- 20%	6 mo.	HNO3
Pb - Lead	200.7	200.2	25 ug/l	1-L poly	1	3	80-120%	+/- 20%	6 mo.	HNO3
Fe - Iron	200.7	200.2	20ug/l	1-L poly	1	3	80-120%	+/- 20%	6 mo.	HNO3
Mg - Magnesium	200.7	200.2	50ug/l	1-L poly	1	3	80-120%	+/- 20%	6 mo.	HNO3
Mn - Manganese	200.7	200.2	2ug/l	1-L poly	1	3	80-120%	+/- 20%	6 mo.	HNO3
Se - Selenium	200.7	200.2	50 ug/l	1-L poly	1	3	80-120%	+/- 20%	6 mo.	HNO3
Ag - Silver	200.7	200.2	10 ug/l	1-L poly	1	3	80-120%	+/- 20%	6 mo.	HNO3
Na - Sodium	200.7	200.2	100ug/l	1-L poly	1	3	80-120%	+/- 20%	6 mo.	HNO3
Zn - Zinc	200.7	200.2	100ug/l	1-L poly	1	3	80-120%	+/- 20%	6 mo.	HNO3
Hg - Mercury	245.1 Cold Vap. AA	245.1	0.2 ug/l	1-L poly	1	3	80-120%	+/- 20%	28 days	HNO3

* Plus 1 duplicate sample for QA out of each 10 drinking water wells (3 triplicate samples) - choose dupe at a well giving highest field indication for NO3

* Plus 1 triple sample for QA out of each 20 lagoon samples (1 triplicate sample)

Table 8 – Manchester Environmental Laboratory – Pesticides

Analyte (9)	Method	Prep Method	Reporting Limit	Container type	Bias (accuracy)	Variability (precision)	Hold time	preservative
Analyte Request								
2,4-D	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen
2,4-DB	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen
Alachlor	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen
Atrazine	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen
Azinphos-methyl	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen
Bromoxynil	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen
Chlorpyrifos	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen

Clopyralid	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen
DCPA	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen
Diazinon	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen
Dicamba	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen
Diclobenil	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen
Dinoseb	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen
Diuron (or breakdown products)	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen
Endosulfan	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen
Fenhexamid	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen
Fenpropathrin	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen

Kresoxim	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen
MCPA	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen
MCPP	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen
Metribuzin	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen
Myclobutanil	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen
Oryzalin	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen
Oxyflruorfen	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen
Pendimethalin	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen
Phosmet	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen
Propargite	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen

Simizine	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen
Terbacil	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen
Triflumizole	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen
Trifluralin	8270D-SIM analysis using 5 - 10uL injections	3535 solid phase extraction 200mg Varian Plexa Cartridge	0.1 - 1 ug/l or 0.1 - 1 ug/kg	2-60ml VOA or 1 8oz. Soil	Liq=70-130% (50-200% Solids)	+/- 30%	28 Days	4 deg. C or solids frozen

	Container	Number per sample	Number needed	Preservation
16 Solids	8 oz soil	1 - 75% full	20 - 8 oz Soil	Frozen -10
46 liquids	60 ml VOA	3 every sample	138 - 60 ml VOA	4deg. C.
Matrix Spike	60 ml VOA	6 - 1 every 20 samples (3)	18 - 60 ml VOA	4deg. C.

Analyst: Randy Cummings
Chemist, USEPA Region 10 Lab
(360) 871-8707

Total VOA 160 - 60ml VOA

Table 9 – Field Parameters

Analyte Specifications for Field Parameters

Field Parameters	Methods from 40 CFR Part 136.3, Table 1B, List of Approved Inorganic Test Procedures:
Turbidity	as NTU, Nephelometric, EPA Method 180.1
Temperature	Degrees C, Thermometric, EPA Method 170.1
Hydrogen Ion	pH Units, Electrometric measurement, EPA Method 150.1
Dissolved Oxygen	mg/L, Electrode EPA Method 360.1
Specific Conductance	micromhos/cm at 25 degrees C, Wheatstone Bridge, EPA Method 120.1
Redox Potential	(mV) Standard Methods 18th Ed. method 2580 B
Nitrate	(by field test kit provided by EPA, as N, mg/L) EPA Method (colorimetric, manual) 352.1
Ferrous Iron	Standard Methods 18th ed., 3500-Fe D", phenanthroline method

Table 10 – Manchester Analytical Laboratory – Microbiology

Dairy Lagoons and Influent Wastewater Treatment Plants:
Method – Standard Method 9221E
Detection Limit 1.8 mpn/100ml
QC will include positive control organisms
Negative control organisms
Transfer Blank (1 per team)
Media Quality Control

Drinking Water Samples:
Method – Standard Method 9222B
Detection Limit 1 cfu/100ml
QC will include positive control organisms
Negative control organisms
Transfer Blank (1 per team)
Media Quality Control

Table 11 –Robert S. Kerr Subsurface Characterization Laboratory, Ada, Oklahoma – Androgen Hormones

Analytes

Compound	Ground Water		CAFO Lagoon	
	MDL (ng/L)	QL (ng/L)	MDL (ng/L)	QL (ng/L)
Estrone	0.07	1	5	40
a-Estradiol	0.16	1	5	40
b-Estradiol	0.12	1	5	40
Ethynyl Estradiol	0.11	1	5	40
Estriol	0.10	1	5	40

This compound also analyzed in UNL Hormone Method

Sample Containers: 1-L and 2-L Pyrex Glass Media Bottles which have been cleaned and fired at 450°C for 4 hr.

Sample Volume: We need 2 L for ground water and drinking water samples and 1 L for lagoons, septic effluent, etc.

Sample Collection: Ideally, samples should be field-filtered through 0.45-µm filters and preserved with formaldehyde. If filtration not possible, denote samples as UNFILTERED. Collect 2 L for ground water and drinking water and add 54 mL of 37% (wt/v) formaldehyde. Collect 1 L for lagoons and septic effluents and add 27 mL of 37% (wt/v) formaldehyde. Note: If lagoons have high alkalinity (> 2000 mg/L CaCO3), formaldehyde addition will cause foaming and gas release. Be sure to mix and vent all pressure before capping bottle. These samples should be kept on ice in large bubble-pak bags.

NOTE: Our teams are not field filtering samples and are not adding formaldehyde.

Table 12 – University of Nebraska – Lincoln – Isotopes of Nitrogen

Analysis

Nitrogen isotope analysis of nitrate and ammonia (^{15}N-NH_4/NO_3) will be determined separately using alkaline distillation of ammonia, Devardas alloy reduction and separate distillation of nitrate as ammonia, followed by oxidation of ammonia to nitrogen gas and dual inlet isotope ratio mass spectrometry (Gormly and Spalding, 1979; Krietler, 1979). Laboratory duplicates, reference standards and blanks will be run at a frequency of 5% of total sample throughput. The measured value of the stable isotope ratios in reference standards will be within 0.5 permil or less of the nominal value in the calibration standards.

we'd prefer to get a frozen 1-liter sample in a plastic bottle (non-filtered is fine).

Sample collection

Use precautions to minimize the potential for contamination of sampling devices. Fill bottles to no more than 70% of the container capacity to allow room for expansion. Store samples in cooler on ice in the field, and then freeze sample to -20oC prior to shipment. Pack in individual zipper bags and wrap with bubble wrap, or other suitable shipping padding, to minimize breakage during transport. Add additional icepacks to prevent melting. Use overnight carrier.

Ship Samples to: Dr. Daniel Snow
202 Water Sciences Laboratory
University of Nebraska-Lincoln
Lincoln, NE 68583-0844

Notify by email dsnow1@unl.edu when shipping samples.

Table 13 –US Geological Survey – Reston Virginia – Dissolved Gases

Sulfur Hexafluoride Age Determination

30 SF6 Samples / 5 Gas Study Samples
2-1 liter glass per sample
60 containers and caps
Store and Ship at ROOM TEMPERATURE in bubble Wrap in Ice Chests
Holding time is 3 months - we will ship as the ice chests become full

To contact us via mail, you may send your message to the following address:

CFC laboratory
US Geological Survey
432 National Center
12201 Sunrise Valley Drive
Reston, VA 20192

Telephone and Fax

Our main telephone number is (703) 648-5847, and our fax number is (703) 648-5832.

Ground water samples are collected in 1 liter glass bottles. The water in-flow tube is placed in the bottom of the bottle displacing the air in the bottle with water. After at least 3 liters of over-flow, the tube is removed. The bottles are capped with Polyseal screw-caps without headspace. Tape the capped bottle with electrical tape. The caps that are not taped come loose during shipping and compromise your samples. The bottles are shipped in coolers to the Chlorofluorocarbon Laboratory in Reston, Virginia for analysis.

Steps for Field Collection of SF_6 Ground Water Samples

1. Purge well
2. Place tubing from pump in the bottom of 1L bottle
3. Fill bottle and allow it to overflow from the neck at least 3 volumes (about 2.5L)
4. Slowly remove tubing from the bottle while water is still flowing
6. Collect two bottles per site.
7. Keep bottles in a cooler but not on ice and not in the sun

Table 14 –US Geological Survey – Denver CO – Trace Organics

Analytes

Analyte Count	Constituent Name	CAS number	Possible Compound Uses or Sources*	Long Term MDL	Reporting Level (QL)	Units
1	1,4-Dichlorobenzene	106-46-7	Moth repellant, fumigant, deodorant	0.02	0.04	ug/L
2	1-Methylnaphthalene	90-12-0	2-5% of gasoline, diesel fuel, or crude oil	0.02	0.04	ug/L
3	2,6-Dimethylnaphthalene	581-42-0	Present in diesel/kerosene (trace in gasoline)	0.06	0.12	ug/L
4	2-Methylnaphthalene	91-57-6	2-5% of gasoline, diesel fuel, or crude oil	0.02	0.04	ug/L
5	3-Methyl-1(H)-indole (Skatole)	83-34-1	Fragrance, stench in feces and coal tar	0.02	0.04	ug/L
6	3-beta-Coprostanol	360-68-9	Carnivore fecal indicator	1	2	ug/L
7	3-tert-Butyl-4-hydroxy anisole (BHA)	25013-16-5	Antioxidant, general preservative	0.3	0.6	ug/L
8	4-Cumylphenol	599-64-4	Nonionic detergent metabolite	0.05	0.1	ug/L
9	4-Nonylphenol diethoxylate, (sum of all isomers) aka NP2EO	NA	Nonionic detergent metabolite	5	5	ug/L
10	4-n-Octylphenol	1806-26-4	Nonionic detergent metabolite	0.08	0.16	ug/L
11	4-tert-Octylphenol	140-66-9	Nonionic detergent metabolite	0.7	1.4	ug/L
12	4-tert-Octylphenol diethoxylate, aka OP2EO	NA	Nonionic detergent metabolite	0.5	1	ug/L
13	4-tert-Octylphenol monoethoxylate, aka OP1EO	NA	Nonionic detergent metabolite	0.5	1	ug/L
14	5-Methyl-1H-benzotriazole	136-85-6	Antioxidant in antifreeze and deicers	1	2	ug/L
15	Acetophenone	98-86-2	Fragrance in detergent and tobacco, flavor in beverages	0.2	0.4	ug/L
16	Acetyl hexamethyl tetrahydronaphthalene (AHTN)	21145-77-7	Musk fragrance (widespread usage) persistent in ground water	0.5	0.5	ug/L
17	Anthracene	120-12-7	Wood preservative, component of tar, diesel, or crude oil, CP	0.02	0.04	ug/L

18	Anthraquinone	84-65-1	Manuf dye/textiles, seed treatment, bird repellant	0.08	0.16	ug/L
19	Benzo[a]pyrene	50-32-8	Regulated PAH, used in cancer research, CP	0.04	0.08	ug/L
20	Benzophenone	119-61-9	Fixative for perfumes and soaps	0.06	0.12	ug/L
21	Bisphenol A	80-05-7	Manuf polycarbonate resins, antioxidant, FR	0.2	0.4	ug/L
***	Bisphenol A-d3	(internal standard)	NA	NA	NA	Pct
22	Bromacil	314-40-9	H (GUP), >80% noncrop usage on grass/brush	0.5	1	ug/L
23	Bromoform	75-25-2	WW ozination byproduct, military/explosives	0.05	0.1	ug/L
24	Caffeine	58-08-2	Beverages, diuretic, very mobile/biodegradable	0.05	0.1	ug/L
***	Caffeine-C13	(internal standard)	NA	NA	NA	Pct
25	Camphor	76-22-2	Flavor, odorant, ointments	0.03	0.06	ug/L
26	Carbaryl	63-25-2	I, crop and garden uses, low persistence	0.5	1	ug/L
27	Carbazole	86-74-8	I, Manuf dyes, explosives, and lubricants	0.02	0.04	ug/L
28	Chlorpyrifos	2921-88-2	I, domestic pest and termite control (domestic use restricted as of 2001)	0.06	0.12	ug/L
29	Cholesterol	57-88-5	Often a fecal indicator, also a plant sterol	1	2	ug/L
30	Cotinine	486-56-6	Primary nicotine metabolite	0.2	0.4	ug/L
31	Diazinon	333-41-5	I, > 40% nonagricultural usage, ants, flies	0.04	0.08	ug/L
32	Fluoranthene	206-44-0	Component of coal tar and asphalt (only traces in gasoline or diesel fuel), CP	0.02	0.04	ug/L
33	Hexahydrohexamethylcyclopentabenzopyran (HHCB)	1222-05-5	Musk fragrance (widespread usage) persistent in ground water	0.3	0.5	ug/L
34	Indole	120-72-9	Pesticide inert ingredient, fragrance in coffee	0.04	0.08	ug/L
35	Isoborneol	124-76-5	Fragrance in perfumery, in disinfectants	0.09	0.18	ug/L

36	Isophorone	78-59-1	Solvent for lacquer, plastic, oil, silicon, resin	0.04	0.08	ug/L
37	Isopropylbenzene	98-82-8	Manuf phenol/acetone, fuels and paint thinner	0.1	0.2	ug/L
38	Isoquinoline	119-65-3	Flavors and fragrances	0.2	0.4	ug/L
39	Menthol	89-78-1	Cigarettes, cough drops, liniment, mouthwash	0.2	0.4	ug/L
40	Metalaxyl	57837-19-1	H, F (GUP), mildew, blight, pathogens, golf/turf	0.06	0.12	ug/L
41	Methyl salicylate	119-36-8	Liniment, food, beverage, UV-absorbing lotion	0.05	0.1	ug/L
42	Metolachlor	51218-45-2	H (GUP), indicator of agricultural drainage	0.04	0.08	ug/L
43	N,N-diethyl-meta-toluamide (DEET)	134-62-3	I, urban uses, mosquito repellent	0.07	0.14	ug/L
44	Naphthalene	91-20-3	Fumigant, moth repellent, major component (about 10%) of gasoline	0.02	0.04	ug/L
45	Pentachlorophenol	87-86-5	H, F, wood preservative, termite control	1	2	ug/L
46	Phenanthrene	85-01-8	Manuf explosives, component of tar, diesel fuel, or crude oil, CP	0.02	0.04	ug/L
47	Phenol	108-95-2	Disinfectant, manuf several products, leachate	0.7	1.4	ug/L
48	Prometon	1610-18-0	H (noncrop only), applied prior to blacktop	0.1	0.2	ug/L
49	Pyrene	129-00-0	Component of coal tar and asphalt (only traces in gasoline or diesel fuel), CP	0.02	0.04	ug/L
50	Tetrachloroethylene	127-18-4	Solvent, degreaser, veterinary anthelmintic	0.06	0.12	ug/L
51	Tributyl phosphate	126-73-8	Antifoaming agent, flame retardant	0.1	0.2	ug/L
52	Triclosan	3380-34-5	Disinfectant, antimicrobial (concern for acquired microbial resistance)	0.1	0.2	ug/L
53	Triethyl citrate (ethyl citrate)	77-93-0	Cosmetics, pharmaceuticals	0.2	0.4	ug/L
54	Triphenyl phosphate	115-86-6	Plasticizer, resin, wax, finish, roofing paper, FR	0.06	0.12	ug/L
55	Tris(2-butoxyethyl)phosphate	78-51-3	Flame retardant	0.4	0.8	ug/L

56	Tris(2-chloroethyl)phosphate	115-96-8	Plasticizer, flame retardant	0.05	0.1	ug/L
57	Tris(dichlorisopropyl)phosphate	13674-87-8	Flame retardant	0.06	0.12	ug/L
58	beta-Sitosterol	83-46-5	Plant sterol	2	4	ug/L
59	beta-Stigmastanol	19466-47-8	Plant sterol	1	2	ug/L
60	d-Limonene	5989-27-5	F, antimicrobial, antiviral, fragrance in aerosols	0.07	0.14	ug/L
61	p-Cresol	106-44-5	Wood preservative	0.09	0.18	ug/L
62	para-Nonylphenol (total) (branched)	84852-15-3	Nonionic detergent metabolite	1	2	ug/L

This compound also analyzed in UNL Mixed Wastewater Pharmaceuticals

*** From Water-Resources Investigations Report 01-4186 - "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory - Determination of Wastewater Compounds by Polystyrene-Divinylbenzene Solid-Phase Extraction and Capillary-Column Gas Chromatography/Mass Spectrometry**

Analytical Sample Request Form must accompany each sample

Description:
1L GCC - This schedule consumes the entire container.
Treatment and Preservation: Baked 1-L amber glass bottle Do not rinse bottle. Fill to shoulder only. Chill. Maintain at 4 deg. C. Ship immediately.

The holding times for LS 1433 and 4433 are 14 days from sampling to sample extraction. We do have some holding time data that has not been published. We recommend that samples are sent to the laboratory overnight chilled at 2-6 degrees Celsius. Treated water should be preserved with ascorbic acid. See WAQI note 07.04 at the following URL: http://water.usgs.gov/usgs/owq/WaQI/WaQI07.04.pdf. Four hours at 400 degrees Celsius is sufficient for cleaning the glassware.

http://pubs.usgs.gov/wri/wri014186/

National Water Quality Laboratory
Building 95, Ent E-3
Denver Federal Center
Denver, CO 80225-0046

Table 15 – University of Nebraska, Lincoln - Steroid Hormone Analyses

Analytes

Compound	MDL (pg/g)	QL (ng/L)
Testosterone	0.3	5
11KetoTestosterone	1.3	5
Androsterone	4.0	5
4-Androstenedione	1.3	5
17a-Hydroxyprogesterone	1.7	5
Progesterone	1.1	5
Melengestrel Acetate	3.2	5
a-Trenbolone	1.7	5
b-Trenbolone	1.6	5
a-Estradiol	1.1	5
b-Estradiol	1.0	5
Estrone	1.6	5
Estriol	1.9	5
Ethynyl Estradiol	1.5	5
a-Zearalanol	4.0	5
a-Zearalenol	3.1	5
b-Zearalenol	2.2	5
b-Zearalanol	3.2	5

This compound also analyzed in Ada Hormone Method

Introduction and Scope

These instructions apply to collection of liquids and solid samples for pharmaceutical and steroid hormone analysis by liquid chromatography-tandem mass spectrometry. Individuals collecting these samples are responsible for ensuring that sample containers and devices used to collect and/or manipulate materials are free of these contaminants.

Sample containers

Because there is no generally accepted method for sample preservation by chemical addition, samples will be frozen as soon as possible to minimize microbial activity after collection. Straight-sided wide mouth amber glass jars (8 or 32oz) allow for expansion of water during freezing and are recommended for sample storage. Eagle Picher (#131-08A, 133-32A), or equivalent.

Sample collection

Use precautions to minimize the potential for contamination of sampling devices. Fill new and pre-cleaned sample jars to no more than 70% of the container capacity to allow room for expansion. Store samples in cooler on ice in the field, and then freeze sample to -20oC prior to shipment. Pack in individual zipper bags and wrap with bubble wrap, or other suitable shipping padding, to minimize breakage during transport. Add additional icepacks to prevent melting. Use overnight carrier.

Ship Samples to:

Dr. Daniel Snow
202 Water Sciences Laboratory
University of Nebraska-Lincoln
Lincoln, NE 68583-0844

Notify by email dsnow1@unl.edu when shipping samples.

Table 16 – University of Nebraska, Lincoln - Veterinary Antibiotic Analyses

Analytes

Compound	MDL (pg/g)	QL (ng/L)
Virginiamycin	1.9	20
Sulfamerazine	14.2	20
Sulfadimethoxine	2.5	20
Sulfamethazole	11.6	20
Sulfathiazole	14.4	20
Sulfachloropyridazine	14.7	20
Sulfamethoxazole	13.7	20
Sulfamethazine	7.5	20
Tetracycline	14.4	20
Oxytetracline	9.7	20
Iso/Chlortetracycline	7.1	20
Tylosin	1.6	20
Lincomycin	11.0	20
Tiamulin	17.7	20
Erythromycin	22.9	20
Monensin	1.0	20
Ractopamine	7.4	20

This compound also analyzed in UNL Mixed Wastewater Pharmaceuticals Method

Ship Samples to:

Dr. Daniel Snow
202 Water Sciences Laboratory
University of Nebraska-Lincoln
Lincoln, NE 68583-0844

Notify by email dsnow1@unl.edu when shipping samples.

Table 17 – University of Nebraska, Lincoln - Mixed Wastewater Pharmaceuticals Analyses

Analytes

Compound	MDL (pg/g)	QL (ng/L)
1,7-Dimethylxanthine	NA	NA
Caffeine	NA	NA
Acetaminophen	NA	NA
Carbamazepine	NA	NA
d-Amphetamine	NA	NA
DEET	NA	NA
Diphenylhydramine	NA	NA
Erythromycin	NA	NA
Ibuprofen	NA	NA
Lincomycin	NA	NA
Methamphetamine	NA	NA
Monensin	NA	NA
Ractopamine	NA	NA
Sulfadimethoxine	NA	NA
Sulfamerazine	NA	NA
Sulfamethazole	NA	NA
Sulfamethoxazole	NA	NA
Sulfathiazole	NA	NA
Thiabendazole	NA	NA
Tiamulin	NA	NA
Tylosin	NA	NA
Azithromycin	NA	NA
Cotinine	NA	NA
Sulfachloropyridazine	NA	NA
Sulfamethazine	NA	NA

This compound also analyzed in USGS Trace Organic Method

This compound also analyzed in UNL Veterinary Antibiotic Method

Table 18 – Cascade Analytical – NO3 in Water and N-Forms in Solids

Cascade Analytical
Lab Union Gap/Wenatchee
Nitrate in ground-water

Analyte	Method	Reporting Limit	Container type	Number of Containers	Container #	Bias (accuracy)	Variability (precision)	Hold time	preservative
Nitrate	300.0	0.04 to 0.3 mg/L	1-L poly	1	1	80-120%	+/- 20%	48 Hours	4deg.C
Total Nitrogen by combustion	AOAC-993.13	<100ppm	1-8oz Soil	1	2	85-115	+/- 20%	14-days	4deg.C
Extractable NO3/NH4	SM-10-107-04-1-A/ Ver4-S-3-10	0.4mg/l	1-8oz Soil	1	3	85-115	+/- 20%	14-days	4deg.C

8.1 Sample Alteration Form

Project Name and Number: _____

Material to be Sampled: _____
_____

Measurement Parameter: _____
_____

Standard Procedure for Field Collection & Laboratory Analysis (cite reference):
_____
_____

Reason for Change in Field Procedure or Analysis Variation:
_____
_____
_____

Variation from Field or Analytical Procedure:
_____
_____
_____

Special Equipment, Materials or Personnel Required:
_____
_____

Initiators Name: _____ Date: _____

Project Officer: _____ Date: _____

QA Officer: _____ Date: _____

8.2 Corrective Action Form

Project Name and Number: _____

Sample Dates Involved: _____

Measurement Parameter: _____
_____

Acceptable Data Range: _____
_____

Problem Areas Requiring Corrective Action:
_____
_____
_____

Measures Required to Correct Problem:
_____
_____
_____
_____

Means of Detecting Problems and Verifying Correction:
_____
_____
_____

Initiators Name: _____ Date: _____

Project Officer: _____ Date: _____

QA Officer: _____ Date: _____

Appendix 1 – SOP for Instrument Calibration

1.0 Scope and Application

This standard operating procedure (SOP) provides guidance for the calibration and use of the Horiba U-53G Multi Water Quality Checker (U-53G) by qualified, field inspectors, investigators, or other field personnel.

2. 0 Summary of Test method

Following guidance from applicable analytical methods, regulations and publications, field personnel prepare a Quality Assurance Project Plan (QAPP) which includes specifications for appropriate field parameters to be measured by the U-53G. Based on the information in the QAPP, a service request is received by Field Support Center personnel and the instrument(s) are prepared and packed for shipment or pickup. Within OEA, selected members of the Environmental Services Unit on-site with Region 10 Manchester Environmental Laboratory provide this service. The U-53G is a muti-meter instrument that can simultaneously measure: pH, Temperature, Dissolved Oxygen, Conductivity, Total Dissolved Solids, Salinity, Turbidity, Oxidation Reduction Potential, Depth and GPS position. The U-53G is composed of the sensor unit, that is lowered into the water, and the hand held monitor, which allow the user to view and log sensor readings. The U-53G is calibrated using the methods below. It is designed for field use and applications include: rivers, lakes, and ground or tap water using a flow through cell.

3.0 Acronyms

COND - Conductivity
DO – Dissolved Oxygen
DOT – United States Department of Transportation
IATA – International Air Transport Association
NIST – National Institute of Standards and Technology
NTU – Nephelometric Turbidity Unit
OEA – Office of Environmental Assessment
ORP – Oxidation Reduction Potential
QA – Quality Assurance
QAPP – Quality Assurance Project Plan
SOP – Standard Operating Procedure
TDS – Total Dissolved Solids
TURB- Turbidity

4.0 Interferences and Precautions

Do not use instrument in magnetic fields. Measurement errors will occur.
Do not immerse in alcohol, organic solvent, strong acid, strong alkaline or other similar solutions.
Does not support measurement of samples containing fluorine.
Do not subject to strong shocks. Do not drop.
Ensure all calibration solutions are the same temperature as ambient air temperature.

Holding the probe while calibrating may cause the internal probe temperature to rise causing a Dissolved Oxygen (DO) calibration error
Do not drop instrument into water lower gently dropping will cause sensors to fail and my produce false readings or instrument failure.
Do not use below depths of 30 meters. Sensor probes are only resistant to 30 meters. Below these depths may give false reading or instrument failure.
Unit must be turned on for 20 minutes prior to calibrating or using DO measurements.
For non-flowing water slowly move instrument up and down to induce flow over DO membrane.
Oxidation Reduction Potential (ORP) standard solutions must be used within one hour at which point it may be transformed. For this reason ORP standard solutions may not be stored.
When measuring ORP with low concentration of oxidants and reductants such as tap water, well water, or water that underwent treatment there may less repeatability and stability, in general.
Always use the calibration cup provided. Other containers may create effects from ambient light that cause incorrect turbidity calibrations.
Avoid turbidity measurement in direct sunlight, since the readout may be affected.

5.0 Safety

Use appropriate personal protective equipment including gloves and safety glasses when using calibration solutions.

6.0 Equipment and Supplies

U-53G multi Water Quality Checker, Control Unit and Sensor Probe
pH Sensor
ORP Sensor
Reference electrode
DO Sensor
Turbidity Sensor

Calibration Cup(s) or appropriate containers as specified in sensor calibration section
pH reference internal solution
DO sensor internal solution set
DO Membrane spare parts set
Spanner wrench for DO sensor
Cleaning Brush
Back pack
Strap
Silicone Grease
Instruction Manuel
De-ionized water (DI water)
Bubbler
pH 4,7,10 Calibration Standard
Sodium Sulfite (zero DO)
Quinhydrone Solution (ORP Standard)
Auto Cal Solutions (0 NTU, pH 4, 4.40 ms/S)
Conductivity Calibration Solutions
Turbidity Calibration Solutions
Sensor Guard and Cap

LR14 alkaline dry cell batteries, C-size
Coin battery, CR-2032
Protective caps for DO, and pH Storage
Flow Cell assembly w/ appropriate size tubing
Scale
Beakers
Graduated Cylinders
Calibration Solution Disposal Container
Calibration Solutions and Standards

pH
pH standard 4, 7, and 10, NIST traceable with expiration date.
Auto Cal Solution, NIST traceable with expiration date

DO
Zero DO solution is a Sodium Sulfite salt mixed with water (DI or Tap) at a ratio of 50g of sodium sulfite to 1000 ml of water.
Span DO solution is water saturated with air. This is done by using a bubbler in a bath of water.
Each solution shall be made fresh just prior to calibration or drift measurement.

ORP
ORP standard powder (quinhydrone) mixed with 250 ml of deionized water and/or premixed ORP standard, NIST traceable with expiration date.

Conductivity
Auto Cal Solution, NIST traceable with expiration date.
Conductivity Standards, range of mS/cm, NIST traceable with expiration date.

Turbidity
Auto Cal Solution, NIST traceable with expiration date.
Turbidity Solutions, Range of NTU's, Auto Cal Solution, NIST traceable with expiration date.

Shipment and Storage

8.1 All equipment and calibration standards or solutions shall be shipped in compliance with the appropriate IATA
or DOT requirements.

8.2 Calibration solutions and standards should be placed in Ziploc bags with taped caps and placed upright in the
shipping container to minimize spilling during shipping.

8.3 If the equipment will be shipped or delivered, it will be placed into ice chests, cardboard boxes, or other
appropriate containers with adequate cushioning material to prevent breakage and custody sealed to confirm not tapering of the standards has take place during shipment.

8.4 Storage For Short Term Use Only

Prior to use inspect DO and pH for sensor caps are present and remove. DO is a white cap, the pH is a black cap.

Replace caps filled with DI water between uses and for short term storage of less than two months. Note: Any water may be used in the field to ensure sensors remain wet. Replace water with DI water once available.

8.5 Follow instruction manual for long term storage, which is defined as storage of longer than 2 months.

9.0 Quality Control and Documentation

Calibration and drift measurements, see section 10, should be conducted for each sampling event at a frequency outlined in the QAPP. For this project, that will be daily prior to field use.

The calibration of the instrument sets each parameter to a given standard or solution.

Drift measurements should also be done at the end of each sampling event. Drift is the measurement of the standards or solutions used for calibration. This measurement will indicate if the instrument has "drifted" during the course instrument use. It is used as an indicator to ensure the probes are in good working order and the readings obtained during the sampling event are accurate.

All calibration solutions and standards shall be made fresh prior to use or be used within the labels expiration date.

All commercial standards or solutions shall be NIST traceable with expiration date.

Documentation of the measurements can occur by two methods: electronically by saving the measurements in the instrument for downloading after the sampling event and/or by logging readings manually into a log book. In either case follow the project specific QAPP or SOP. For example: Inspection type activities require a logbook with numbered pages and must be bound.

An equipment log book accompanies the U-53G. Each user shall enter User name, Location of equipment use, Date equipment used, Calibrations that occurred, and other applicable information or failures. This log book remains with the U-53G and should not be used as the data log for the project.

10.0 Calibration and Standardization

The U-53G offers two methods to calibrate. Selection of the calibration method depends on how accurate and the quality of data needed. For general screening the Auto Calibration generally will meet the needs. The second method each parameter is manually calibrated with a zero and a span(s) standard. It is the operator's responsibility to know the limitations of this instrument, any EPA standard methods that apply, and the quality of data required by the project and/or QAPP. Operators should become familiar with the instruction manual for the U-53G. This outline will not duplicate the instruction manual verbatim.

Auto Calibration

PRIOR TO USE REMOVE DO AND pH SENSOR STORAGE CAPS
Turn unit on using the power button. For DO sensors the unit must remain on for 20 minutes to stabilize and prior to calibration.
Using the auto cal solution and fill calibration cup to the "with TURB" line located on the side of the cup.
Place calibration cup into black calibration cup to eliminate light sources.
Press the CAL button or right arrow to the Calibration Tab.
Press the down arrow to Auto Calibration
Press Enter and follow instructions on screen.
Instructions should then direct you to place sensors into calibration cup.
Monitor values of the sensors once stabilized press ENTER to start calibration
Do not remove the unit from calibration solution the will display "-----" until finished.
The display will direct you to press MEAS to exit to measurement screen.
If error occurs make note of error code and refer to instruction manual, Section 4.6 Troubleshooting.

Manual Calibration

Manual Calibration is used to individually calibrate sensors. It is used to ensure good measurement precision throughout all measurement ranges. It should be noted the temperature calibration should follow the most current version of the Inorganic Chemistry Support Equipment Monitoring SOP along with procedures outlined below, since the H-53G allows temperature calibration the temperature obtained from the Inorganic Chemistry Support Equipment Monitoring SOP can be entered directly into the H-53G and no correction factor is necessary.

PRIOR TO USE REMOVE DO AND pH SENSOR STORAGE CAPS

Turn unit on using the power button. For DO sensors the unit must remain on for 20 minutes to stabilize and prior to calibration.

Press the CAL button or right arrow to the Calibration Tab.

Temperature (Performed yearly or as needed)

Following the Inorganic Chemistry Support Equipment Monitoring SOP
Press the CAL button or right arrow to the Calibration Tab.
Press the down arrow to Manual Calibration
Move the cursor to Temp and press ENTER
Press the up and down arrow to adjust value
Once value is stable press ENTER to start calibration
When calibration is finished press ENTER for measurement mode

pH

Calibrate the zero point (pH 7) first, rinse calibration cup 2-3 times with DI water, then fill with pH 7 standard solution to the w/TURB reference line.

Rinse the senor probe 2-3 times with DI water to remove any dirt and then submerge into calibration cup

Press CAL or from the Calibration Tab and press the down arrow to "Manual Calibration"
Move the cursor to pH and press ENT
Set the number of calibration points for two
Press the up and down to set the pH value of 7.00 for the pH 7 standard, reference temperature vs. pH standard value chart
Check that the value has stabilized then press ENTER to start calibration.
Once complete press Enter to continue to span cal.
Rinse calibration cup 2-3 times with DI water
Fill cup with pH 4 or pH 10 to the w/TURB reference line. Place sensor into calibration cup.
Check that the value has stabilized, reference temperature vs. pH standard value chart, press ENTER to start calibration.
When calibration complete press ENTER to return to calibration parameter menu.

ORP

Rinse the sensor probe and calibration cup 2 to 3 times with DI water
Place sensor probe into solution.
Fill calibration cup with ORP standard to the w/TURB reference line.
Press the units CAL key to set calibration mode
Press down arrow to Manual Calibration and press ENTER
Move the cursor to ORP and press ENTER
Press the up or down arrow to adjust the mV to match the standard mV for the given standard temperature.
Check the measurement value has stabilized and press ENTER
Once complete press ENTER to return to the calibration selection screen.

Conductivity (COND)

Rinse the sensor probe 2 to 3 times with DI water to remove any dirt
Remove as much moisture from the sensor, the zero calibration is done in air.
Press the units CAL key to set calibration mode
Press the down arrow to Manual Calibration and press ENTER
Move the cursor to COND and press ENTER
Press up or down arrow to select number of calibration points.
Press up or down arrow to set COND to 0.00 mS/cm
Check the measurement value has stabilized and press ENTER
Once complete press ENTER to proceed to first span calibration procedure
Wash the sensor probe and calibration cup 2 to 3 times with DI water.
Fill calibration cup to the w/TURB reference line with a 0.00 to 0.999mS/cm standard.
Place sensor probe into solution.
Press up or down to set value to standard.
Check to ensure measurement value has stabilized and press ENTER
Once complete press ENTER to proceed to the second span calibration procedure.
Wash the sensor probe and calibration cup 2 to 3 times with DI water.

Fill calibration cup to the w/TURB reference line with a 1.00 to 9.99 mS/cm standard.
Place sensor probe into solution.
Press up or down to set value to standard.
Check to ensure measurement value has stabilized and press ENTER
Once complete press ENTER to proceed to the third span calibration procedure.
Wash the sensor probe and calibration cup 2 to 3 times with DI water.
Fill calibration cup to the w/TURB reference line with a 10.0 to 100.0 mS/cm standard.
Place sensor probe into solution.
Press up or down to set value to standard.
Check to ensure measurement value has stabilized and press ENTER
Once complete press ENTER to proceed to the calibration parameter screen.

Turbidity (TURB)

Rinse the sensor probe and calibration cup 2 to 3 times with DI water
Fill calibration cup with zero standard to the w/TURB reference line.
Place calibration cup into black calibration cup to eliminate light sources.
Place the sensor probe into calibration cup with standard.
Press the units CAL key to set calibration mode
Press down arrow to Manual Calibration and press ENTER
Move the cursor to TURB and press ENTER
Press the up or down arrow to select number of calibration points
Press up or down to set value to the 0.00 standard.
Check to ensure measurement value has stabilized and press ENTER
Once complete press ENTER to proceed to the first span calibration procedure.
Wash the sensor probe and calibration cup 2 to 3 times with DI water.
Fill calibration cup to the w/TURB reference line with a 0.1 to 10.0 NTU standard.
Place sensor probe into solution.
Press up or down to set value to standard.
Check to ensure measurement value has stabilized and press ENTER
Once complete press ENTER to proceed to the second span calibration procedure.
Wash the sensor probe and calibration cup 2 to 3 times with DI water.
Fill calibration cup to the w/TURB reference line with a 10.0 to 100.0 NTU standard.
Place sensor probe into solution.
Press up or down to set value to standard.
Check to ensure measurement value has stabilized and press ENTER
Once complete press ENTER to proceed to the third span calibration procedure.
Wash the sensor probe and calibration cup 2 to 3 times with DI water.
Fill calibration cup to the w/TURB reference line with a 100 to 1000 NTU standard.
Place sensor probe into solution.
Press up or down to set value to standard.
Check to ensure measurement value has stabilized and press ENTER
Once complete press ENTER to proceed to the calibration parameter screen.

Dissolved Oxygen (DO)

Prepare standard solutions

Add 50 g of sodium sulfite to 1000 ml of water and stir to dissolve. Generally, 1000 ml of solution is not needed for one calibration. Use the ratios 12.5 per 250 ml or 25 g per 500 ml depending on number of instruments that will be calibrated.
Pour 1 to 2 liters of water into a suitable container and feed air into water using a bubbler or air pump until it is oxygen saturated.
Rinse the sensor probe 2 to 3 times with DI water
Place sensor into zero standard into the calibration cups (black and clear). Fill solution over the DO sensor slot in the cup, this is the area between the black cup and the clear cup.
Press the units CAL key to set calibration mode.
Press down arrow to Manual Calibration and press ENTER.
Move the cursor to %DO and press ENTER.
Press the up or down arrow to selecting two (2) for the number of calibration points.
Press the up or down arrow to adjust the %DO to match zero.
Check to ensure the measurement value has stabilized and then press ENTER.
Once complete press ENTER to proceed to span calibration procedure.
Place sensor guard on sensor unit to protect sensors.
Place sensor probe into oxygen saturated bath solution.
Press up or down to set value to 100%.
Check to ensure measurement value has stabilized and press ENTER.
Once complete press ENTER to return to the calibration selection screen.

Water Depth (DEPTH)

Rinse the sensor probe 2 to 3 times with DI water
Press the CAL button or right arrow to the Calibration Tab.
Press the down arrow to “Manual Calibration”
Move the cursor to Depth and press ENTER.
Press the up and down arrow to adjust value to 0.00 meters
Once value is stable press ENTER to start calibration
When calibration if finished press ENT for measurement mode

Drift Measurements

Turn unit on using the power button. For DO sensors the unit must remain on for 20 minutes to stabilize and prior to calibration.

Using the calibration cup or appropriate container with each successive standard or solution and measure the value by pressing the MEAS button.

Record and compare to the standard or solution value. The difference is the drift value.

A percentage of drift can be calculated by subtracting the drift measurement from the standard and dividing by the standard value. This percentage can be used to monitor the equipments sensors and to ensure data quality and repeatability.

Measurement and Data Collection

Any instrument requests should be made to Field Services staff via phone or email with at least two weeks notice to ensure adequate standards are available and equipment is serviceable.

Prior to use remove DO and pH sensor storage caps

Single Measurement

Turn unit on using the power button. For DO sensors the unit must remain on for 20 minutes to stabilize, use the internal clock and DO reading, and prior to calibration.
Check that each sensor and sensor guard is mounted.
Press the right or left arrow to the Single Measurement tab or screen.
NOTE: DO NOT press MEAS key while unit in the air, this will cause turbidity wiper failure.
Submerge sensor probe in the sample, gently shake them to remove air bubbles form sensors.
For non-flowing water gently move sensor probe up and down at a rate of 20 to 30 cm a second.
When non-turbidity values are stable press MEAS to acquire values.
Press ENTER to save the values or write values in logbook.
Press ESC to cancel operation.
Continue to next measurement or press and hold PWR button to power off.

GPS Information

Press the right arrow to the Data Operations tab or screen.
Press the down arrow to move the cursor to GPS Information then press ENTER.
When position data exists it will be displayed, if no data is displayed data has not been received.
Data Operations

Press the right arrow to the Data Operations tab or screen.
Press the down arrow to move the cursor to View Data then press ENTER.
Move the cursor to Site/Date/All and then press ENTER. The Site name or Date will need to be entered depending on your selection. All data will display with the most recent first.
Press the up or down arrow to scroll though data.
To delete data from the Data Operation tab or screen cursor to Delete Data and press ENTER.
Press the left arrow to confirm Yes and press ENTER. All data will be deleted.
It is the operator's responsibility to save data obtained during the sampling event. The unit may be cleared of all data upon return either by the ESU personnel or the next user.

Calibration Record Check

Press the right arrow to the Data Operations tab or screen.
Press the down arrow to move the cursor to Calibration Record then press ENTER
The latest calibration record will be displayed. Scroll by pressing the down arrow

Sensor Information

Press the right arrow to the Information tab or screen.
When sensor probe is normal "All sensors are available." will appear.
When a sensor has a problem a message will be generated. Refer to the instruction manual for troubleshooting information. (Section 4.6 pages 89 to 94)

Calculations

Principals of measurements for each parameter are outlined in the instruction manual for the instrument. Refer to pages 100-109 or Sections 6.3 thru 6.11

Some of the calibrations solutions need to be mixed. Refer to the calibration section of the instruction manual or section 10 above for mixture ratios.

Refer to appropriate calibration chart for standard value for a given temperature. Usually located on the bottle and/or U-53G instruction manual. There may be a need to interpolate between temperature readings to obtain the correct standard value.

Percentage of Drift = (Calibration Standard – Drift Measurement) / Calibration Standard

Waste Management and Pollution Prevention

Recycle used containers when possible both in the field and in accordance with Manchester Environmental Laboratory's Waste Management and Pollution Prevention Programs when at the Manchester Lab Facility. Disposal of calibration solution should only occur in an appropriate manner, i.e. not on the ground or in a body of water. All waste solutions should be saved and disposed of once you have returned from the field.

11.0 References

Horiba U-50 series, Multi Water Quality Checker, Instruction Manual, April 2009.
Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), current version.
Standard Methods for the Examination of Water and Wastewater, Joint Editorial Board, American Public Health Association, American Water Works Association and Water Environment Federation, latest edition.
Code of Federal Regulations, Title 40 (40 CFR), Part 136.3, Table IB.
Pollution Prevention Plan of the Manchester Environmental Laboratory,@ USEPA Region 10,current version.
Manchester Environmental Laboratory Dangerous Waste Disposal Manual,@ USEPA Region 10, current version.

Appendix 2 – SOP for Shipping Container Preparation

U.S. Environmental Protection Agency, Region 10
Office of Environmental Assessment
Maja Tritt, CLP PO, 205.553.6265
Bethany Plewe, RSCC, 206.553.1603
Jennifer Crawford, RSCC, 206.553.6261

R10 Manchester Environmental Laboratory (MEL)
Lab Support Work Flow

1. Submit 2 copies of QAPP to RSCC and QA reviewer 3-4 weeks prior to sampling. Contact those above for assistance in preparing the QAPP. If modifications are required to the CLP SOW (ex: low detection limits) submit QAPP 4 weeks prior to sampling (electronic version is acceptable).
2. Use assigned Regional sample numbers and project code on all sample labels and TR/COCs.
3. RSCC supplies copies of FORMS II Lite software and custody seals.
4. Contact RSCC with shipping information via email (preferred) or **on the day that you ship**. Include the following information:
   Project code
   Airbill number
   Date shipped, date to arrive at lab
   Laboratory shipped to
   Number of samples including matrices (not number of bottles)
   Analysis needed on samples, note samples for QC
   Note when case is complete
5. MEL is closed after 4:30 pm on weekdays and closed on weekends.
6. Electronic, validated analytical data are expected to be sent to EPA and contractor PMs within 8 weeks or turnaround time described by MEL, after lab receipt of samples
7. For all superfund, CLP or non-CLP work, submit TR/COCs (.xml format) to the ESDS website immediately after shipping is completed.
   http://epasmoweb.fedcsc.com/scstr/
   Username: R10ESDS
   Password: uploadR10
   **include analytical costs or email them to Elizabeth Holman (holman.elizabeth@epa.gov)
8. Submit the Regional copy of ALL TR/COCs for every project to the RSCC when the last samples are shipped.
9. REMINDER: Submit TR/COCs to lab, RSCC, and ESDS

USEPA REGION 10
SAMPLE CONTAINER REQUIREMENTS FOR MEL

SHIPPING SAMPLES

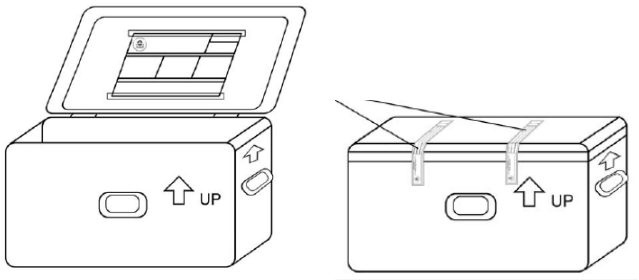
Conduct an inventory of the contents of the shipping cooler/container against the corresponding TR/COC record. Wrap each glass bottle with plastic bubble wrap. Use suitable non-combustible packing materials (ex. air filled plastic bags) for the coolers. Vermiculite and kitty litter packing materials are not allowed.

Cover samples with double-bagged ice (bottom, sides and top). Do not pour loose ice directly into the sample cooler unless each of the sample containers is sealed in a plastic bag.

Include a 40-ml vial filled with water and labeled "**temperature blank"** with each cooler shipped. Do not assign a sample number for the temp blank.

Place signed TR/COCs and return cooler label in a ziplock plastic bag and tape it to the lid of each cooler.

Include necessary paperwork = signed TR/COCs, sample labels, return cooler label.



USEPA REGION 10
BOTTLE CODE REQUIREMENTS FOR MEL

In order for the Region 10 Manchester Laboratory to remain NELAC certified, they have to be able to track every bottle that is received. Region 10 uses container codes in order to track every bottle. One sample number can consist of several containers from one location for a variety of

analyses. 2 digit alphanumeric codes are assigned to each bottle for each sample number. The first digit in the code refers to the preservative used in the sample, and the second digit refers to the bottle count associated with the preservative and sample number.

Preservative Codes
A - HCl
B - HNO_3
C - NaOH
D - H_2SO_4
E - $Na_2S_2O_3$
F - ascorbic acid, then HCl
G - $Na_2S_2O_3$ + EDTA
H - EDTA
N - No chemical preservation
P - Bottles per-preserved at lab
T - To be preserved at the lab

Example 1: If you are collecting a soil sample with EPA number 08184000 from location to be analyzed for SVOCs and total metals, you'll have 3 containers (2-8oz jars for SVOC and QC, 1-4oz jar for total metals and QC). No chemical preservation is necessary, so for sample #08184000, you'll have 3 container codes associated with it = N1, N2, N3 each respectively indicated on each container.

Example 2: If you are collecting a water sample with EPA number 08184001 from a location to be analyzed for SVOCs and total metals, you'll have 7 containers (6-1L amber glass for SVOC and QC, and 1-1L HDPE bottle for total metals and QC). No chemical preservation is necessary for the SVOCs, but HNO3 preservation is required for total metals in water. The container codes for the 7 bottles associated with #08184001 are N1, N2, N3, N4, N5, N6, B1

Appendix 3 –Job Hazard Assessment for Sampling Team

OFFICE/UNIT/PROGRAM: OEA	**DATE COMPLETED: 4-8-2010**
JOB/TASK: SET UP LOGISTICS FOR WELL SAMPLING STUDY	**PROJECT LOCATION: YAKIMA VALLEY, WA**
SUPERVISOR(S): CURT BLACK	**ANALYSIS PERFORMED BY: CATHE BELL / CURT BLACK**
DATE(S) TASK TO BE PERFORMED: APRIL 12 – APRIL 22, 2010	**TYPE OF TASK:** ☒One time ☒ Routine task

TO COMPLETE THE FOLLOWING CHART, DESCRIBE THE ASPECT OF THE WORK YOU PERFORM WHICH YOU JUDGE MOST HAZARDOUS. IDENTIFY THE STEPS TAKEN TO ACCOMPLISH THAT WORK, THE HAZARDS ASSOCIATED WITH EACH STEP, AND MEANS OF CONTROLLING OR LIMITTING THOSE HAZARDS.

ACTIVITY SEQUENCE	ENVIRONMENTAL, SAFETY, OR HEALTH HAZARD	HAZARD CONTROLS
1. Prepare and load materials for sampling activity.	Lifting, twisting, low back strain. Forgetting materials.	Careful lifting techniques, secure grip, packing at desk level or higher. Lists.
2. Drive to sampling area.	Motor vehicle crash, snow, ice, lost of traction, weather, pass conditions, driving in unfamiliar areas and conditions.	Select rested, experienced driver. down in adverse conditions. Read driving guidance (appendix x). Co with laws including Executive Orde
3. Check into post in Yakima area.	Material transfer, storage. Unsuitable residence.	Careful lifting technique, park close assure good footing, wear appropri clothing including protective foot ge
4. Review plans for day's activities.	Failure to be thoroughly prepared.	Engage in logistics meetings and g communication. Review problems day before considering controls to prevent a repeat.
5. See JHA: Household tap water sampling.	See JHA: Household tap water sampling.	See JHA: Household tap water sampling.
6. Pool and pack samples for delivery to lab/FedEx..	Weight, bending, twisting, lifting, dropping: both the samplers and receivers are at risk.	Careful lifting techniques. Secure Careful, padded packing at desk le higher. Do not pack boxes too hea
7. Drive to west side of mountains.	MVA, adverse weather conditions, visibility, see #2.	Refer to map prior to leaving. Be attentive and responsive to local d conditions. See 2.
8. Drop samples, supplies.	Weight, lifting, carrying.	Park close. Stretch on exiting vehi Careful lifting techniques. Secure

HAZARDS –NOTE ALL POTENTIAL HAZARDS ASSOCIATED WITH THE WORK ACTIVITY.

Physical

Radiation	☐ ionizing	☐ microwave	☒ light
	☐ heat	☒ cold	☒ noise
	☐ explosion	☐ fire	
Vehicles	☒ traffic	☐ heavy equip	☐ forklift
	☐ helicopter	☐ small aircraft	☐ boat
Boat Ops	☐ sampling	☐ rapid water	☐ open water
	☐ diving	☐ electrofish	☐ weather
Industrial	☐ comp gas	☐ electricity	☐ conf space
	☐ equip	☐ moving parts	
Overhead	☐ obstruction	☐ falling objs	
Elevation	☐ roof	☐ scaffold	☐ ladder
	☒ stairs	☐ catwalk	
Slips/trips	☒ terrain	☒ debris	☒ slippery
	☐ trench	☒ pits/holes	

Other physical hazards: ☐

Biological

	☒ fatigue	☒ violence
Agriculture	☐ CAFO	☐ fish
Animals	☒ dogs	☐ feral animals
Insects	☒ spiders	☐ mosquit
	☐ bees	
Pathogens	☒ bloodborne	☒ sewage
Other Biological:		☒ TB/MAR

Chemical

Containers	☐ ammonia	☐ chlorine
VOCs	☐ solvents	☐ fuel
Wastes	☐ sewer	☐ landfill
	☐ metals	☐ PCBs
Particulates	☐ fibers	☐ diesel
Sampling	☒ acids	☐ bases
		package
Other Chem:		☒ samples

Comments:

PERSONAL PROTECTIVE EQUIPMENT (PPE) REQUIRED (CIRCLE ALL THAT APPLY)

PE

Feet:	x	safety shoes	safety-toe		shank
		rubber boots	waders		
Gloves:		leather	cotton		cut-resistant
		chem resist	x disposable	x	warmth
Body:	x	safety vest	pfd		harness
		tyvek	sarnex-tyvek		coveralls
Eyes:	x	safety glasses	sunglasses		goggles
Head:		hard hat	hearing prot		respirator

OTHER SAFETY EQUIPMENT/CONSIDERATIONS

Other Safety Equipment and Procedures

			Communication		decon
		dosimetery	x fire extinguisher		flares
x		first aid kit		x	
x		chains/studs	wash		

Other Equipment/Procedures:

Emergency Contact:

Other PPE:	
REMINDERS:	
• have clear agency identifications at all times • wear seatbelts, drive at the speed limit • know your emergency numbers and evacuation routes • violence is unacceptable: pull back and get help • sanitize after contact with dirty water (alcohol gel or wipes)	• know your limitations / take breaks when needed • stay hydrated – bring water supplies with you • be able to stay dry (carry extra socks, etc.) • wear sunscreen (SPF 15 or higher) and / or hats when needed • remember biological hazards (repellant)
CERTIFICATION OF HAZARD ASSESSMENT	
EPA FIELD SAMPLING MANAGER: CURT BLACK **DATE:4-8-2010**	**SAFETY/HEALTH REPRESENTATIVE: CATHE BELL** **DATE: 1/26/2010**

Task Hazard Analysis Form

I HAVE READ OR BEEN BRIEFED ON THE HAZARDS AND PROTECTIVE MEASURES IDENTIFIED FOR THE ABOVE-LISTED TASKS AND FULLY UNDERSTAND THE TASK-SPECIFIC REQUIREMENTS THAT HAVE BEEN ESTABLISHED.			
DATE	**EMPLOYEE NAME**	**EMPLOYEE SIGNATURE**	**EMPLOYER NAME**

OFFICE/UNIT/PROGRAM: OEA	**DATE COMPLETED:1-26-2010**
JOB/TASK: HOUSEHOLD TAPWATER SAMPLING-YAKIMA NITRATE STUDY	**PROJECT LOCATION: YAKIMA VALLEY, WA**
SUPERVISOR(S): CURT BLACK	**ANALYSIS PERFORMED BY: CATHE BELL/CURT BLACK**
DATE(S) TASK TO BE PERFORMED: FEB 22-MAR 7, 2010	**TYPE OF TASK:** ☒One time ☐ Routine task

TO COMPLETE THE FOLLOWING CHART, DESCRIBE THE ASPECT OF THE WORK YOU PERFORM WHICH YOU JUDGE MOST HAZARDOUS. IDENTIFY THE STEPS TAKEN TO ACCOMPLISH THAT WORK, THE HAZARDS ASSOCIATED WITH EACH STEP, AND MEANS OF CONTROLLING OR LIMITTING THOSE HAZARDS.

ACTIVITY SEQUENCE	ENVIRONMENTAL, SAFETY, OR HEALTH HAZARD	HAZARD CONTROLS
Drive to sampling site. Park.	Driving hazards, navigating unfamiliar areas,	Plan your trip, review the days stops, assign a navigator, park so as to depart without

Task Hazard Analysis Form

Exit vehicle, contact homeowner/tenant, unload sampling gear.	Mistaken for evil-doer or Immigration, greeted with prejudice, injury during lifting, yard may be cluttered, slippery walk or steps in poor repair	Always work in teams, don't look sneaky, wear bright colors, have someone on team in EPA logo
Bring sampling gear to house.	Keep outside dirt outside, cluttered house, indoor air quality, bio-hazards in sink area, dogs, nervous home owner	WATCH YOUR STEP, TRANSFER TO INSIDE SHOES FROM OUTSIDE BOOTS, WATCH FOR TRIPPING HAZARDS, USE
Go to well, survey position with GPS, note pipe diameter, condition of sanitary seal, other observations.	Moving in unfamiliar yard, more potential for dog interactions, neighbors, decreased situational awareness while focused on GPS or in well house, black-widow spiders in well house	Ask home owner about dogs, have a light, watch your step, watch your surroundings, don't' penetrate into well house more than necessary for observations of well condition
Enter house. Arrange sampling gear.	Homeowner upset over mud inside home, difficult working conditions,	Keep outside dirt outside – switch shoes at the door, CREATE AN ORGANIZED WORK AREA, SPREAD
Prepare for sampling: disassemble kitchen spigot, clean spigot, plumb spigot into multiparameter cell.	Sink may need to be emptied for flow-through cell, knives or sharps in sink, water may be outgassing H2S or contain coliform bacteria or pathogenic organisims.	ISOLATE YOURSELF FROM THE CONTENTS OF THE SINK, OR MOVE THE CONTENTS WITH CARE , SANITIZE WORKING AREA
Collect samples.	Splash contact with pathogens, if TKN sample is needed, acid handling for preservation,	Work neatly, avoid splashing and producing aerosols, before opening acid, put on eye protection,
Stop sampling. Reconnect water delivery.	Sharp corroded plumbing, splash hazard	Carefully replace aerator on faucet – if it breaks, place the aerator in a Ziploc bag with the address of the
Pack samples and sampling equipment.	Injury from lifting ice chests full of equipment,	Clean and dry equipment, pack the acid container if used in a protective container
Exit house, carrying samples and equipment to vehicle.	same tripping hazards as during entry, lifting injuries from heavy loads, same dogs as during entry	Watch your step, lift properly and organize in multiple smaller loads. Demobe and leave the home
Load car.	Injury from lifting and twisting during packing of vehicle	Lift correctly, keep loads manageable, get help with packing
Drive to next site.	Risks of driving, dark, icy roads, unfamiliar roads, manure trucks, feed trucks, milk trucks, cattle trucks, Vacuum Trucks,	Plan your trip, review the next stop, assign a navigator, watch for trucks, carry chains, use the most

Task Hazard Analysis Form

HAZARDS –NOTE ALL POTENTIAL HAZARDS ASSOCIATED WITH THE WORK YOU HAVE PLANNED .	

Task Hazard Analysis Form

Physical

Radiation	☐ ionizing	☐ microwave	☐ light
	☐ heat	☒ cold	☐ noise
	☐ explosion	☐ fire	
Vehicles	☒ traffic	☒ heavy equip	☐ forklift
	☐ helicopter	☐ small aircraft	☐ boat
Boat Ops	☐ sediment sampling	☐ rapid water	☐ open water
	☐ diving	☐ electrofish	☐ weather
Industrial	☐ comp gas	☐ electricity	☐ conf space
	☐ equip	☐ moving parts	
Overhead	☐ obstruction	☐ falling objs	
Elevation	☐ roof	☐ scaffold	☐ ladder
	☐ stairs	☐ catwalk	
Slips/trips	☒ terrain	☒ debris	☒ slippery
	☐ trench	☒ pits/holes	
Other physical hazards:		☒ DOGS	

Biological

	☐ fatigue	☒ violence	☒ illness/injury
Agriculture	☐ CAFO	☐ fish	☒ farm animals
Animals	☒ dogs	☒ feral animals	☒ snakes
Insects	☒ spiders	☐ mosquitoes	☐ wasp/hornet
	☒ bees		
Pathogens	☒ bloodborne	☒ sewage	☐ med/lab
Other Biological:		☒ TB, MARSA	

Chemical

amm chlo o

Task Hazard Analysis Form

PERSONAL PROTECTIVE EQUIPMENT (PPE) REQUIRED (CIRCLE ALL THAT APPLY)				
PPE				
Feet:		safety shoes	safety-toe	shank
	x	rubber boots	waders	
Gloves:		leather	cotton	cut-resistant
		chem resist	x disposable	
Body:	x	safety vest	pfd	harness
		tyvek	sarnex-tyvek	coveralls
Eyes:	x	safety glasses	sunglasses	goggles
Head:		hard hat	hearing prot	respirator

OTHER SAFETY EQUIPMENT/CONSIDERATIONS					
Other Safety Equipment and Procedures					
			Communication		decon
		dosimetery	fire extinguish		flares
x		first aid kit	x	h x	Hand sanitizer
x		chains/studs	eye wash	h x	
Other Equipment/Procedures:					

Emergency Contact:

Other PPE:

REMINDERS

- have clear agency identifications at all times
- wear seatbelts
- drive at the speed limit
- know your emergency numbers and evacuation routes
- violence is unacceptable: pull back and get help
- sanitize after contact with dirty water (alcohol gel or wipes)
- know your limitations / take breaks when needed
- stay hydrated – bring water supplies with you
- be able to stay dry (carry extra socks, etc.)
- wear sunscreen (SPF 15 or higher) and / or hats when needed
- remember biological hazards (repellant)

CERTIFICATION OF HAZARD ASSESSMENT

EPA FIELD SAMPLING MANAGER: CURT BLACK

DATE: 1/26/2010

SAFETY/HEALTH REPRESENTATIVE: CATHE BELL

DATE: 1/26/2010

Task Hazard Analysis Form

OFFICE/UNIT/PROGRAM: OEA	**DATE COMPLETED:**
JOB/TASK: DAIRY/CROPLAND/LAGOON SAMPLING	**PROJECT LOCATION: YAKIMA COUNTY**
SUPERVISOR(S): CURT BLACK	**ANALYSIS PERFORMED BY: CATHE BELL/ CURT BLACK**
DATE(S) TASK TO BE PERFORMED: VARIOUS	**TYPE OF TASK:** ☐One time xx☐ Routine task

TO COMPLETE THE FOLLOWING CHART, DESCRIBE THE ASPECT OF THE WORK YOU PERFORM WHICH YOU JUDGE MOST HAZARDOUS. IDENTIFY THE STEPS TAKEN TO ACCOMPLISH THAT WORK, THE HAZARDS ASSOCIATED WITH EACH STEP, AND MEANS OF CONTROLLING OR LIMITTING THOSE HAZARDS.

ACTIVITY SEQUENCE	ENVIRONMENTAL, SAFETY, OR HEALTH HAZARD	HAZARD CONTROLS
1. Stock chests (ice, sample containers)	Lifting, twisting, sprain, strain, drop	Buddy, assist each other. Careful lifting techniques. Use small coolers rather than large ones
2. Collect and load sampling gear Electric pump sampler Long pole sampler	Weight, lifting carrying, contaminate gear, tripping, poking, awkward movements	"" Use a dolly or lifting mechahism, a mechanical assist; maintain assured clear distance
3. Transport staff and sampling materials to site	Road hazards, driving skills, other vehicles/drivers, vehicle capability, weather, soft driving surfaces off road and in lots.	Select confident driver; study map before departure; get information on parking locations from operator prior to arrival; use 4-wheel drive if necessary. Inspect vehicle for gas/oil/tire pressure/spare tire/ready inflate/jack/safety flares/water/radio; defensive driving techniques; avoid distractions; use seat belts; drive attentively; comply with laws. Allow someone to rideshotgun with map and advice.
4. Gain access to the site	Interpersonal tensions, farm animals, dogs, denial of access, electric fences, slips, immersions, solid surfaces over liquid, suffocation. Biohazard cross-contamination across sites.	Be aware of farm animals, the cattle are precious. Avoid bulls. Avoid or be introduced to dogs who may accompany you. Never turn your back

Task Hazard Analysis Form

5. Be aware of operations on facility	Facility may have truck traffic, tractors, heavy equipment and personnel who are not used to visitors in unusual places or conducting sampling	Be aware of facility operations, watch for equipment and personnel and stay visible and make sure you are noticed. Attempt to get your work done efficiently and disturb the operation of the facility as little as possible while still collecting the necessary samples.
5. Take sampling supplies to sampling location	Snags, wet-surfaces, surfaces which appear solid but are liquid underneath. Losing balance, slip into liquids, awkward movements, exposure to methane which displaces air and ammonia at exposure limits. Liquids may contain Insecticides, antibiotics, growth hormones, bacteria. Mosquitoes both as irritants and disease vectors (W. Nile Virus, E Equine encephalitis, etc.), flies	“”Buddy, assist each other. Careful lifting techniques. Carry only materials needed for the sampling activity and team support. Wear long sleeves and tight weave “carhart”- type work clothing or coveralls. Insect repellant. Avoid areas where ventilation is not assured or compromised (manure holding pits, lagoons bound by high walls). Medical countermeasures: tetanus, communication regarding chronic diseases and
6. Set up field work station	Lack of balance, lifting, dropping sampling materials, potential for contamination, insect irritants/vectors, heat/cold sun;	“” Do not eat at sampling location. Disinfectant wipes and hand cleaner.
7. Take samples	“ “ Slope into lagoon, irregular surfaces, lagoon liquid, per se. Extended reach, sampling load carried far from center of gravity (potential to lose control or use excess force to control), contamination from spills,	“” Use rope of harness if slope is extreme. If water soluable (odorous) gases are noticed and irritating, move upwind to access sampling location.
8. Store samples	Lifting, twisting, sprain, strain, drop, weight, carrying, contaminated gear, awkward movements	Careful lifting techniques. Follow sample and hand decontamination process.
9. Reiterations of steps 5-8	Reiterations of steps 5-8	Reiterations of steps 5-8
10. Return materials to vehicles	See 5. Increased weight, contaminated gear.	See 5. Break apart loads making multiple trips if advisable. Assist each other. Careful lifting techniques. Avoid areas where ventilation is not assured or compromised.
11. Package and send to lab	Weight, lifting strains to carriers and lab shipment receivers.	Careful loading, No packages over 35 pounds.

Task Hazard Analysis Form

HAZARDS –NOTE ALL POTENTIAL HAZARDS ASSOCIATED WITH REFERENCED WORK.		

Task Hazard Analysis Form

Physical

Radiation	☐ ionizing	☐ microwave	☒ light
	☒ heat	☒ cold	☐ noise
	☐ explosion	☐ fire	
Vehicles	☒ traffic	☒ heavy equip	☐ forklift
	☐ helicopter	☐ small aircraft	☐ boat
Boat Ops	☒ sampling	☐ rapid water	☐ open water
	☐ diving	☐ electrofish	☒ weather
Industrial	☐ comp gas	☒ electricity	☐ conf space
	☐ equip	☐ moving parts	
Overhead	☒ obstruction	☐ falling objs	
Elevation	☐ roof	☐ scaffold	☐ ladder
	☐ stairs	☐ catwalk	
Slips/trips	☒ terrain	☒ debris	☒ slippery
	☒ trench	☒ pits/holes	

Other physical hazards: ☐

Comments:

Biological

				violence	☒
	☒	fatigue	☒		
Agriculture	☒	CAFO	☐	fish	☒
				feral animals	☒
Animals	☒	dogs	☐		
				mosquitoes	☒
Insects	☐	spiders	☒		
	☐	bees			
				sewage	☐
Pathogens	☐	bloodborne	☒		

Other Biological: ☐

Chemical

				chlorine fuel	☐
Containers	☐	ammonia	☐		

Task Hazard Analysis Form

PERSONAL PROTECTIVE EQUIPMENT (PPE) REQUIRED (CIRCLE ALL THAT APPLY)							OTHER SAFETY EQUIPMENT/CONSIDERATIONS		
PPE							**Other Safety Equipment and Procedures**		CG-meter
Feet:	x	safety shoes		safety-toe	x	shank		x	meter
	x	rubber boots		waders					dosimetery	x	Communic
						cut-	x	first aid kit		fire extingu
Gloves:		leather		cotton		resistant			chains/studs	x	eye wash
		chem resist	x	disposable		double	Other Equipment/Procedures: x			
Body:		safety vest		pfd	x	harness				
				sarnex-						
		tyvek		tyvek	x	coveralls				
Eyes:		safety glasses	x	sunglasses		goggles	**Emergency Contact:**			
Head:		hard hat		hearing prot		respirator				
Other PPE:										

REMINDERS:

- have clear agency identifications at all times
- wear seatbelts
- drive at the speed limit
- know your emergency numbers and evacuation routes
- violence is unacceptable: pull back and get help
- sanitize after contact (alcohol gel or wipes)
- know your limitations / take breaks when needed
- stay hydrated – bring water supplies with you
- be able to stay dry (carry extra socks, etc.)
- wear sunscreen (SPF 15 or higher) and / or hats when needed
- remember biological hazards (repellant)

CERTIFICATION OF HAZARD ASSESSMENT

SUPERVISOR/CERTIFIER: CURT BLACK C

DATE: APRIL 8, 2010

SAFETY/HEALTH REPRESENTATIVE: CATHE BELL

DATE: APRIL 8 2010

I HAVE READ OR BEEN BRIEFED ON THE HAZARDS AND PROTECTIVE MEASURES IDENTIFIED FOR THE ABOVE-LISTED TASKS AND FULLY UNDERSTAND THE TASK-SPECIFIC REQUIREMENTS THAT HAVE BEEN ESTABLISHED.

DATE	EMPLOYEE NAME	EMPLOYEE SIGNATURE	EMPLOYER NAME

Task Hazard Analysis Form

Appendix 4 - GPS SOP and Instructions

Garmin GPSmap 60CSx

Equipment Check List
- GPS unit
- Owner's Manual
- AA batteries

MapSource software – install on your computer.
PC-download cable – USB mini-B plug to A-type plug
Learn button and screen functions

Appropriate area maps have been uploaded into the units
We've loaded Metroguide 7 for Washington, Oregon, and Idaho into the units.
Main Menu (Press "MENU" button twice)
- Setup
  - System
    - GPS – Normal
    - WAAS – Enabled
    - Battery Type – Alkaline or NiMH
    - External Power Lost – Stay On
  - Time
    - Time Format – 12 or 24 Hour
    - Time Zone – ENTR correct time zone
    - Daylight Saving Time – Auto
  - Units
    - Position Format – hddd.ddddd°
    - Map Datum – NAD83
    - Distance/Speed – Statute
    - Elevation – Feet

Operation
1. After turning the unit on, wait until it has completed "Acquiring" and/or "Locating" Satellites and that you have "3D" coverage.
2. Note your Location accuracy estimate. Under good conditions it should indicate better than +/- 30 ft. If the accuracy is acceptable for the intended purpose, record your position. If not, wait for the accuracy to improve, or move to a different location. Satellites constantly move into and out of view, so it may not be a long wait.
3. Recording your position.
   a. Press the "MARK" button.
   b. Record the waypoint number in field notes;
   c. To record an average of multiple satellite fixes, after noting the waypoint number, scroll the cursor to "Avg" and press the "ENTR" button. The number of satellite fixes is indicated on the bottom of the screen as "Measurement Count." Once you've reached approximately 60 fixes (abouyt 1 minute) , press the "ENTR" button to "save" your averaged position, then highlight "OK" and press the "ENTR" button again. Note: 60 measurements are usually adequate.
   d. Press the "QUIT" button to exit the waypoint screen.

4. Pressing the “PAGE” button allows you to switch between various screen modes.
5. Pressing the “MENU” button once shows specific menu options for the current page/screen.
6. Pressing the “MENU” button twice from any screen takes you to the Main Menu options.

Accessing Previously Logged Waypoints

1. Press the “FIND” button.
2. Highlight “Waypoints” on the screen, and press the “ENTR” button.
3. Highlight the waypoint of interest and then press the “ENTR” button. This displays the location and options to delete, show on map, or navigate.

Appendix 5 – Custody Forms and Laboratory Specific Tracking Forms

EPA EPA Region 10, 1200 Sixth Avenue, Seattle, WA 98101

Sample Custody & Analysis Required Form

EPA Manchester Laboratory, 7411 Beach Drive East, Port Orchard, WA 98366, 360-871-8700

Form Effective Date: July 2005

Revision 1

Project Name	*Project Code*	*Method of Shipment/carrier*	*Airbill Number (if known prior to sealing):*
Account Code	*EPA Project Manager/phone number*	*Check all that apply* ☐ Enforce/Custody ☐ Possible Toxic/Hazardous ☐ Data Confidential	

Sampler Names (Print & Sign). *Mark* (R) *after name of principal recorder.*

If applicable, circle the set of selected metals:

Al	Sb	As	Ba	Be
B	Cd	Ca	Cr	Co
Cu	Fe	Pb	Mg	Mn
Mo	Ni	K	Se	Ag
Na	Sn	Tl	V	Zn

(see reverse for more to add/circle)

① Matrix Codes:
10 Water/Liquid (Total)
20 Water/Liquid (Filtered)
40 Sediment/Soil/Solid/Bulk
70 Tissue
80 Oil/Solvent
44 Air filter
42 Wipe/Swab[1]
00 _____

[1] PCB wipe is to be 10cm x 10cm (100 cm²)

#C ② enter the number of containers for each preservative type followed by the appropriate preservation code P ③:

A - HCl
B - HNO_3
C - NaOH
D - H_2SO_4
E - $Na_2S_2O_3$
F - ascorbic acid[2], then HCl
G - $Na_2S_2O_3$+EDTA
H - EDTA
N - No chemical preservation
P - Bottles pre-preserved at lab
T - To be preserved at the lab

[2] $Na_2S_2O_3$ *if required by plan.*

W - _____

☐ Check here if the cooler is iced

④ Enter the letter or range of letters on each container for each group of containers with the same preservative type. Each container for each unique sample number must have a unique letter on it.

Sampler's comments for the laboratory:

Laboratory: *see the applicable QAPP, SOW and/or Analytical Support Request for specific methods and detection, reporting, and/or quantitation limits*

EPA Sample number Yr	Wk	Sequence	Sampling Date & Time Yr	Mo	Day	Time	Matrix ①	#C ②	P ③	#C ②	P ③	#C ②	P ③	#C ②	P ③	Sampler Initials	Sample/Station Description/Field Measurements	Organics (see reverse) VOA	BNA	Pest	PCB	PAH	Metals (see reverse) CLP	Selected	Mercury	Micro (see reverse) E. Coli	T. Coliform	General Chemistry (see reverse) TSS	TDS	BOD 5	NO3+NO2	Oil & Grease	Asbestos	Additional Write In Analyses (see reverse)			

Chain of Custody Record

Relinquished by *(Signature)*	Date	Time	Received by *(Signature)*	Date	Time
Relinquished by *(Signature)*	Date	Time	Received by *(Signature)*	Date	Time
Relinquished by *(Signature)*	Date	Time	Received by **Mobile Lab** for Field Analysis *(Signature)*	Date	Time
Shipped by *(Signature)*	Date	Time	Received for lab by *(Signature)*	Date	Time

Receiving Laboratory Information Condition of Samples upon Receipt at Lab:

Custody Seals Intact: ☐ yes ☐ no ☐ none

Distribution: White - Laboratory Copy;
Yellow - Regional Sample Control Center (RSCC) Copy; Pink - Field or Office Copy

Task Hazard Analysis Form

Andrew Greenwood, Shaw Environmental,
USEPA KERR LAB
919 Kerr Research Dr.
Ada, OK 74820
(580)436-8920

ANALYTICAL SAMPLE RECORD

(For samples to be submitted to Shaw Env.)

Technical Directive #: 6MD114ED

Project Name: Yakima Estrogen Samples

For Shaw Env. use only — Sample Received on: _____ at: _____ Received by: _____
Log-in by: _____ Date: _____ Sample STAD Set Number: _____

SAMPLE INFORMATION: (**Matrix:** W = Water, S/S = Soil/Sediment, O = Others; **Container types:** G = Glass, P = Plastic, O = Others)

ANALYSES / PARAMETERS (Number of containers and type, e.g., 2G, 3P, etc.)

#	Sample I.D.	Sample Collection Date	Sample Collection Time	Sample Matrix (use the codes)	Total Number of Containers	ESTROGENS								Comments
1				W	1G	1G								
2				W	1G	1G								
3				W	1G	1G								
4				W	1G	1G								
5				W	1G	1G								
6				W	1G	1G								
7				W	1G	1G								
8				W	1G	1G								
9				W	1G	1G								
10				W	1G	1G								
11				W	1G	1G								
12				W	1G	1G								
13				W	1G	1G								
14				W	1G	1G								
15				W	1G	1G								
16				W	1G	1G								
17				W	1G	1G								

Task Hazard Analysis Form